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WHEAT AND FLOUR STUDIES, VII
MILLING AND BAKING TESTS OF FROZEN AND NON-FROZEN WHEAT HARVESTED AT VARIOUS STAGES OF MATURITY[1]

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Introduction

Wilhoit (1916), Birchard (1920) and Whitcomb, Day, and Blish (1921) have reported results obtained on submitting frosted wheat to the baking test. In general, the flour from badly frosted immature wheat is of poor quality and would not be suitable for bread making. If the frost has affected the wheat only slightly, such as a light blistering of the bran layer, and there is no appreciable amount of green kernels, the baking value of the wheat seems to be unimpaired.

Most of the previous work has been done with wheat frosted in the field. The factors which may affect the quality of frosted wheat under field conditions are so numerous and variable that it is very difficult to recognize their individual effects. The low baking quality of badly frosted wheat may be due to any one of several changes which freezing might produce or initiate. In our investigation, we have tried to reduce the number of variable factors as much as possible.

In the most of our work we have made freezing conditions so severe that if the freezing produced no change in the factors under consideration, we could be reasonably sure that milder freezing conditions to which wheat would be subjected in the field would not influence them. On the other hand, if severe freezing temperatures were found to affect these factors, the next step would be, logically, to study the effect of various so-called degrees of frost.

[1] Published with the approval of the Director.

Experimental

The milling was done with an Allis-Chalmers experimental mill with two stands of break rolls and one of reduction rolls. The first break rolls had 14 corrugations per inch, the second had 20 corrugations.

The wheat was first cleaned to free it of all foreign material. A 1500-gram portion was then scoured in an automatic scourer to remove all dust and loose particles of bran. Sufficient water was added to the scoured wheat to bring the moisture content up to 14.5%. After stirring, the wheat was allowed to stand from 36 to 48 hours before milling.

The wheat was then passed twice through the coarse break rolls and sifted through the 20-wire sieve. The portion remaining on the sieve was ground in the fine break rolls three times, sifting the first time with the 24-wire sieve, and the second and third times with the 26-wire sieves. The portion remaining on the 26-wire sieve was weighed as bran, and that passing through the various wire sieves was separated and reduced, as later described, to obtain the flour.

This stock was separated into sizings and first, second, third, and fourth middlings, and break flour, by means of silk bolting cloth of the following sizes: 30xx, 40xx, 50xx, 70xx, and 10xx. The sizings and the first and second middlings were passed through an air-blast purifier to remove all light particles of bran. These various separations of stock were then ground on the reduction rolls and sifted each time to remove the shorts as the process proceeded. In this sifting process, the 10xx cloth was replaced with 12xx, through which all stock passed before it was considered as flour. This reduction process was continued to correspond, as nearly as possible, in yield of straight flour produced, with the test weight per bushel of wheat as shown by the diagram in Service and Regulatory Announcements, Markets No. 62, United States Department of Agriculture, May 1920. The percentage of straight flour was computed on the basis of the weight of clean wheat before scouring.

It was found that this method of milling was fairly rapid and produced a straight flour of uniform quality.

The flour was made into bread by a standard formula which produced uniform results and showed the strength of the various

flours. The ingredients in the baking process were as follows:

Flour	340 grams
Sugar	15 "
Salt	5 "
Yeast	10 "
Lard	1 teaspoon
Water	According to absorption

The flour, sugar, and salt were weighed out and placed in the proofing cabinet at 32°C. over night. Water at 32°C. and yeast were added to the sugar and salt in beakers and allowed to ferment for 30 minutes. A Fleischmann dough mixer was used, the mixing period being 2 minutes. The melted lard was added at the time of mixing. The dough was placed in a graduated expansion tube and allowed to ferment at 32°C. until it attained maximum expansion. It was then removed and folded six times and replaced to allow it to treble its volume, after which it was molded into loaves and placed in tall narrow baking pans, 3½x7 inches at the top with sloping sides so that the bottom was 2¾x6¼ inches, and 5½ inches deep.

The dough was allowed to proof at 32°C. until it attained its maximum volume without showing weakness on top. The time elapsing from the mixing of the dough to placing in the oven varied from 135 to 180 minutes. The loaf was baked at 200°C. for 30 minutes.

After cooling 30 minutes, the loaf was weighed and its volume determined by the displacement of vetch seed in an "hour glass" measuring device. The following day the loaves were cut, and scored for color and texture of the cut surface, and the hydrogen-ion concentration was then determined. Three check loaves were baked each time to test the uniformity of the yeast and other conditions. These check loaves were used as a basis for scoring color and texture of the test loaves.

Freezing of Dough

Doughs containing varying amounts of water were subjected to freezing temperatures. Similar doughs were allowed to stand the same length of time in the icebox and the proofing cabinet. Freezing was accomplished by placing the doughs in gallon buckets and leaving them outdoors over night at a temperature of about —15°F. The doughs were frozen solid and the next morning were thawed out and the yeast, sugar, and salt were added.

Methods of treatment and baking data are given in Table I, which is self-explanatory. We may conclude from these experiments that simply freezing the dough does not change the chemical or colloidal properties in such a manner as to destroy the baking quality of the flour. The effect of mechanical treatment on the dough is shown in test 18, Table I. The dough had lost 74 grams during the mixing. This amount of water was added to the dough to replace the loss. The yeast was added at the end of the mixing.

TABLE I

INFLUENCE OF FREEZING ON BAKING QUALITY OF DOUGH AND THE EFFECT OF EXCESSIVE MECHANICAL TREATMENT

Yeast, sugar, and salt were added after the treatment. Each treatment refers to the flour for one loaf.

Lab. No.	Treatment	Absorption	Fermentation time	Loaf volume	Texture	Hydrogen-ion conc.
		%	Min.	cc.	%	pH
1	Check	66	210	1968	100	5.21
4	25 cc. of water added as vapor. Stood in proofing cabinet over night.	59	213	1812	96	5.21
5	Dough made with 210 cc. H_2O. Stood in proofing cabinet over night.	66	225	1908	98	5.26
6	Dough made with 210 cc. H_2O. Stood in icechest over night.	63	209	2045	100	5.29
7	Same as No. 6.	63	204	1973	100	5.32
8	25 cc. water added as vapor. Frozen.	66	192	1878	95	5.33
9	Same as No. 8	66	215	1728	95	5.31
10	50 cc. water added and stirred in as thoroly as possible. Frozen.	62	195	1567	90	5.30
11	100 cc. water to make dough. Frozen.	59	220	2021	98	5.31
12	Flour cooled and 70 gm. of snow shaken up with it, warmed up. Frozen.	57	210	1944	96	5.26
13	150 cc. of water to make dough. Frozen.	65	237	1800	95	5.41
14	180 cc. of water to make dough. Frozen.	62	202	2009	100	5.31
15	200 cc. of water to make dough. Frozen.	62	207	2009	100	5.31
16	210 cc. of water to make dough. Frozen.	63	210	1841	96	5.34
17	Same as 16.	63	197	1943	96	5.30
18	Mixed for 1 hr. Not frozen.	62	210	981	55	5.25

Freezing of Soaked Wheat

To follow the effect of freezing a step nearer to field conditions, a sample of Marquis wheat was divided into aliquots. Some of these were soaked in distilled water at room temperature for various periods of time. After soaking, aliquots of wheat were spread out to air dry at room temperature, while other aliquots were placed outdoors for 48 hours, with the mean outside temperature close to 0°F. After the period of freezing, these aliquots were

also spread out to air dry. The wheat was milled and the flour baked. The results are given in Table II. The soaked wheat samples, whether frozen or not, all yielded larger loaves than the flour from the original wheat. In the 8-hour and the 48-hour soaking experiments, the flour from the frozen wheat gave a lower loaf volume than that from the non-frozen wheat. It should be noted that loaf 5 was of coarse texture, indicating some abnormality.

TABLE II
Influence of Freezing on Soaked Wheat

Lab. No.	Hours soaked in water	Moisture content	Treatment before drying	Loaf volume	Texture	Color
		%		cc.	%	%
Untreated	0	....	....	1662	97	95
1	8	35.7	Not frozen	1878	97	96
2	8	35.7	Frozen	1770	96	95
3	18	39.7	Not frozen	1800	96	95
4	18	39.7	Frozen	1821	96	95
5	48	43.4	Not frozen	1944	90	95
6	48	43.4	Frozen	1716	97	95
Check	....	....	....	1866	100	100

Note: R. E. Kellogg made the baking tests reported in Tables I and II and also milled the samples of wheat for the bakings reported in Table II.

The frozen and non-frozen samples which were soaked 18 hours gave practically the same loaf volume. The conclusion drawn from this experiment was that freezing had no injurious effect upon the baking properties of the flour and that soaking apparently caused an improvement. This improvement would be expected because the wheat was a good sample of Marquis. The kernels were corneous and the protein content was 14.76 per cent. It apparently contained plenty of protein of good quality, but lacked in diastase.

Freezing of Immature Heads of Wheat

In the experiments next described the wheat was frozen at various stages of growth and compared with samples gathered at the same time but not frozen.

Most of the investigators who have studied the baking value of wheat harvested at various stages of maturity have cut the wheat, leaving the straw attached. It is known that in such cases the kernel will continue to develop to a certain extent, owing to the transfer of material from the straw to the kernel. This investigation was planned to study the effect of harvesting wheat at various stages of maturity, using samples of wheat in which the

development was stopped as soon as possible after cutting. The most practical way of accomplishing this seemed to be to harvest only the heads and allow them to dry. This procedure did not prevent all changes after harvest, but it reduced the supply of food material to a greater extent than if the straw had been attached.

At two- to four-day intervals, heads from a field of Marquis spring wheat of the 1923 crop were harvested and half of the heads were at once spread on the floor of a large, well ventilated room to dry. The other half were placed in the hardening room of an ice cream manufacturing plant for about 48 hours and then spread on the floor to dry. The temperature of the hardening room ranged from —20° C. to —28° C. After air drying, the wheat was threshed and milled and the flour baked. Some of the analytical data on this wheat are given by Sharp (1925) and the germination tests by Whitcomb and Sharp (1925). The later article also shows a picture of the kernels at four stages of development.

Data which are of interest in considering the milling and baking value of this series are given in Table III.

TABLE III

FROZEN (F) AND NON-FROZEN (NF) MARQUIS WHEAT OF THE CROP OF 1923 HARVESTED AT VARIOUS STAGES OF MATURITY

Lab. No.	Approx. age of kernel	Moisture in kernels at time of harvest	Vacuum dry wt. per kernel	Protein in wheat (dry basis)	Test weight per bu.	Density of air-dry wheat
	Days	%	mgm.	%	Lbs.	
131 NF	13	69.4	7.68	16.13	40.0	1.4180
132 F	13	69.4	9.15	14.59	46.5	1.4245
133 NF	17	62.5	14.04	15.02	48.4	1.4167
134 F	17	62.5	13.88	14.49	53.3	1.4310
135 NF	21	56.2	19.50	15.40	56.8	1.4180
136 F	21	56.2	20.57	15.19	59.3	1.4307
137 NF	25	50.6	25.28	15.77	61.7	1.4171
138 F	25	50.6	24.50	15.10	60.7	1.4268
139 NF	27	46.5	25.67	16.19	62.2	1.4176
140 F	27	46.5	25.66	16.18	60.2	1.4208
141 NF	29	46.5	26.77	17.00	62.5	1.4163
142 F	29	46.5	27.53	17.29	60.4	1.4236
145 NF*	33	43.5	29.89	16.73	63.4	1.4157
146 F	33	43.5	29.07	17.08	61.6	1.4179
147 NF	35	38.7	30.67	17.08	63.2	1.4168
148 F	35	38.7	30.85	16.70	61.3	1.4179
149 NF	38	34.1	30.19	16.98	63.5	1.4135
150 F	38	34.1	32.66	17.06	62.4	1.4127
151 NF	41	.....	31.01	17.81	63.3	1.4153
152 F	41	.....	32.07	17.03	63.3	1.4123
153 NF	53	9.3	.....	16.65	62.8	1.4156
155 NF†	.....	.....	.....	17.08	63.5	1.4204

*Main part of field harvested at this stage.
†Sample taken from the shock.

TABLE IV

MILLING AND BAKING DATA ON FROZEN (F) AND NON-FROZEN (NF) MARQUIS WHEAT, 1923 CROP, HARVESTED AT VARIOUS STAGES OF MATURITY
Milled Dec. 27, 1923. Baked Feb. 7, 1924.

Lab. No.	Percentage of mill products: Bran	Percentage of mill products: Shorts	Flour: Break	Flour: Total	Milling loss	Protein in the flour (dry basis)	Dry gluten	Absorption	Fermentation time	Loaf volume	Percentage of volume of check	Color	Texture	Hydrogen-ion concentration of bread
	%	%	%	%	%	%	%	%	Min.	cc.	%	%	%	pH
131 NF	19.1	30.2	5.5	34.2	16.5	15.07	....	...	...	....	...	...	...	....
132 F	15.6	20.1	4.4	57.8	6.5	14.60	....	...	...	....	...	...	...	....
133 NF	17.1	19.4	5.1	56.5	7.0	14.39	....	...	...	....	...	...	...	....
134 F	12.1	18.1	3.4	66.7	3.1	14.47	....	...	...	....	...	...	...	....
135 NF	15.7	18.5	6.6	63.4	2.3	14.62	12.5	67	144	1790	87	80	82	5.86
136 F	10.7	18.5	2.8	70.8	0.0	15.27	12.1	69	127	1380	67	30	50	5.86
137 NF	13.9	12.7	9.4	71.2	2.2	15.10	13.9	62	140	1910	93	90	85	5.74
138 F	12.0	20.7	3.5	66.8	0.5	14.47	12.5	66	136	1580	77	80	60	5.64
139 NF	13.6	13.5	9.3	70.4	2.5	15.62	14.5	62	140	1960	96	90	85	5.75
140 F	11.4	19.2	5.9	68.2	1.2	15.47	14.4	64	126	1970	96	88	98	5.74
141 NF	13.8	12.8	9.4	71.5	1.9	15.91	14.7	60	134	1800	88	96	95	5.79
142 F	10.9	14.5	8.2	73.4	1.2	17.14	15.3	60	139	1970	96	96	98	5.75
145 NF*	13.2	11.6	10.2	72.5	2.7	16.08	15.1	58	151	1890	92	96	95	5.74
146 F	10.8	16.3	8.1	71.2	1.7	16.37	14.9	58	152	2030	99	98	98	5.70
147 NF	13.6	14.4	10.8	69.3	2.7	16.39	15.8	58	157	2050	100	98	98	5.74
148 F	11.2	14.2	8.1	73.5	1.1	16.07	15.7	58	167	2020	99	99	100	5.67
149 NF	13.7	10.9	10.5	73.4	2.0	16.42	15.9	59	159	2090	102	96	98	5.75
150 F	13.7	10.9	9.9	72.7	2.7	16.09	15.4	60	156	2130	104	99	100	5.70
151 NF	15.1	11.5	10.1	71.4	2.0	16.47	15.3	58	155	2090	102	99	98	5.71
152 F	13.7	12.9	10.1	71.5	1.9	16.02	14.9	56	165	2090	102	98	98	5.67
153 NF	14.1	12.4	10.4	74.0	+0.5	15.82	15.3	60	160	2060	100	98	98	5.70
155†	15.1	10.9	10.6	71.4	2.6	15.96	14.9	58	155	2020	99	96	85	5.72
Check average of four	....	....	....	....	..	....	....	..	185	2050	100	100	100	5.41

*Main part of field harvested.
†Taken from the shock.

Of special interest is the relationship between the weight per measured bushel and the density of the wheat. In the samples up to No. 135 and 136, the frozen kernels have the greatest weight per measured bushel, and from that point on the lesser weight. It is believed that this lesser weight is due to the blisters on the kernels, which would keep the kernels farther apart and thus give less weight. The frozen kernels actually have a greater density up to samples 149 and 150. On the whole, the density of the non-frozen samples is fairly uniform throughout the period of development studied, while the density of the frozen samples decreases slightly.

The wheat samples were milled twice. The first milling was done December 27, 1923, and the flour was baked February 7, 1924. The data obtained are given in Table IV. February 22, 1924, another baking was carried out on the flour from this milling. The idea has been expressed by some investigators that the beneficial effect of aging is due to the increase in acidity. In repeating the baking on this later date, 2 cc. of normal lactic acid was added to each loaf. The results are given in Table V. Approximately one year after the first milling and baking, the wheat samples were again milled and baked. The data obtained are given in Table VI.

TABLE V

MARQUIS WHEAT 1923 CROP

Milled Dec. 27, 1923. Baked Feb. 22, 1924.

Two cubic centimeters of a normal solution of lactic acid was added to each dough at the time of mixing. No lactic acid was added to the check.

Lab. No.	Absorption	Fermentation time	Loaf Vol.	Percentage of vol. of check	Color	Texture	Hydrogen-ion concentration of bread
	%	Min.	cc.	%	%	%	pH
135 NF	67	135	1670	82	91	86	5.61
136 F	69	146	1300	64	30	40	5.66
137 NF	62	150	1930	95	91	86	5.50
138 F	66	150	1490	73	50	40	5.38
139 F	62	162	2000	98	93	90	5.48
140 F	64	147	1950	96	91	90	5.51
141 NF	60	158	2030	100	95	94	5.51
142 F	60	146	2040	100	95	94	5.49
145 NF	58	170	2060	101	95	96	5.48
146 F	58	178	2090	102	95	94	5.43
147 NF	58	178	2080	102	97	98	5.43
148 F	58	183	2230	109	98	98	5.41
149 NF	61	183	2120	104	98	98	5.50
150 F	60	187	2190	107	98	98	5.51
151 NF	58	178	2090	102	98	98	5.48
152 F	56	174	2190	107	98	98	5.43
153 NF	60	175	2140	105	98	96	5.43
155 NF	58	168	2090	102	93	90	5.51
Check average		202	2040	100	100	100	5.35

TABLE VI

Milling and Baking Data on Frozen (F) and Non-Frozen (NF) Marquis Wheat, 1923 Crop
Milled Dec. 29, 1924. Baked Jan. 9, 1925.

Lab. No.	Test weight per bu.	Yield of flour	Dry gluten	Absorption	Fermentation time	Loaf volume	Percentage of volume of check	Color	Texture	Hydrogen-ion concentration of bread
	lbs.	%	%	%	Min.	cc.	%	%	%	pH
135 NF	56.3	66.8	13.0	70	156	2200	101	92	92	5.74
136 F	58.9	72.3	11.8	72	145	1480	68	40	35	5.69
137 NF	61.0	72.7	13.6	66	135	2010	93	94	94	5.65
138 F	60.3	73.1	11.2	68	134	1730	80	65	50	5.59
139 NF	61.6	73.5	13.9	65	137	1900	88	94	94	5.63
140 F	60.1	74.3	13.4	64	136	2000	92	92	92	5.66
141 NF	61.7	71.6	14.5	60	155	2130	98	96	97	5.59
142 F	60.1	72.5	14.7	60	144	2070	95	96	94	5.62
145 NF	63.0	75.7	14.8	62	148	1950	90	94	95	5.61
146 F	61.1	71.9	15.0	62	145	2300	106	96	97	5.64
147 NF	62.9	74.5	15.4	61	168	2360	109	96	97	5.62
148 F	60.5	77.5	15.3	61	160	2320	107	96	97	5.60
149 NF	63.3	75.3	15.2	60	166	2200	101	96	96	5.63
150 F	61.8	73.9	15.7	60	154	2300	106	96	97	5.55
151 NF	63.0	75.1	15.8	61	155	2260	104	96	97	5.64
152 F	62.5	76.0	15.4	60	165	2370	109	96	97	5.63
153 NF	62.7	74.7	15.2	61	166	2250	104	96	97	5.66
155 NF	63.1	74.9	15.7	62	172	2230	103	95	94	5.66
Check	....	....	....	......	227	2170	100	100	100	5.31

In milling the frozen wheat, it was observed that less break flour and bran were produced and more of the offal came out in the shorts stream than was the case with the non-frozen sample at the same stage of maturity. These points are made clear by an examination of Table IV. The amount of wheat from samples 131 to 134 available for milling ranged from 200 to 500 grams. For this reason the mill fractions of these samples are subject to great error, which probably explains why the first four samples do not agree with the above statement.

The color and texture of the bread baked from the immature samples of frosted wheat were very low. There was, however, a marked improvement with development and from samples 142 on, the texture and color were about the same in the frozen and the non-frozen samples. Probably the loaf volume is the best indication of the quality of the proteins present. In order to make the loaf volume data more comprehensible, they are presented graphically in Figure 1. At those stages of development where the moisture content of the kernel at the time of the freezing was greater than about 46 per cent, the loaf volume, color, and texture were affected unfavorably. If, however, the moisture content was less than this amount, the freezing apparently had no detrimental influence on the quality of the flour. It should be borne in mind that the freezing in these experiments was produced by lower temperatures than the wheat would encounter in the field and consequently, under less severe freezing conditions, the wheat might be uninjured for baking when it contained a still higher moisture content at the time of freezing. On the other hand, freezing in the field might cause changes to take place when the ripening was finished in the field which would produce wheat of lower quality. The data in Figure 1 indicate that if the moisture content is 46 per cent or less at the time of freezing the freezing has no detrimental effect on the loaf volume, and the loaf obtained would be the same as that given by non-frozen wheat at the same stage of maturity. The data also indicate that there is a slight increase in baking quality of wheat as it matures. If the samples of frozen wheat gathered the 27th day are compared with those gathered the 41st day, it is seen that frozen wheat at this immature stage is not equal in baking quality to wheat harvested at the later stage, but the non-frozen wheat at this immature stage is also of slightly lower baking quality. Figure 1 indicates that after the 27th day the frozen samples give slightly larger loaves than the non-frozen samples. However, the data are not sufficient to

prove this point. It will be shown in a later paper that the frozen samples contained more sugars than the non-frozen samples at the same stage of maturity. The data as a whole seem to justify the statement that freezing after the moisture content has been reduced below 46 per cent does not injure the baking quality of the wheat, and that any lower baking quality evidenced by wheat frozen at this moisture content or less is due to the immaturity of the wheat and not to the freezing. This statement is true, provided the changes which take place in the field after freezing are not different from those which took place after the freezing as carried out in our experiment.

The hydrogen-ion concentration of the baked bread was determined electrometrically. The determinations were made on solutions obtained by extracting 8 grams of bread with 25 cc. of distilled water, with occasional shaking, for one hour. The determinations were made the next day after baking. A slight decrease in the pH of the baked bread is indicated after aging the wheat for one year, as shown by a comparison of Tables IV and VI. The addition of lactic acid decreased the pH of the baked bread. This is shown by a comparison of Tables IV and V. If we consider the samples from the 27th day on, and compare them on the basis of the effect of acid and age on the loaf volume, we can interpret our data in two ways, depending on whether or not we consider the actual loaf volumes or the percentage loaf volume, as compared with the standard. The average loaf volume of the non-frozen wheat samples of the first milling was 1980 cc.; of the frozen samples 2035 cc., or 55 cc. in favor of the frozen wheat. This difference is probably not so large, for in Figure 1 A we see that the loaf volumes for the non-frozen samples of the 29th and 33rd days are probably too small. If we consider the volumes of the frozen and the non-frozen loaves as the same at these two points, we have the respective averages of 2032 cc. for the non-frozen and 2035 cc. for the frozen or, on the basis of percentage of standard loaf, approximately, the values are 99.1 per cent and 99.3 per cent respectively. After the addition of lactic acid the loaf volume was increased. The average increase, if we consider the points 29 and 33 in error, is 31 cc. and 80 cc. for the non-frozen and frozen respectively, or a percentage increase on the basis of the standard of 1 per cent and 4.4 per cent respectively.

If we consider the effect of aging the wheat approximately one year, the volumes are 2133 cc. and 2227 cc. for the non-frozen

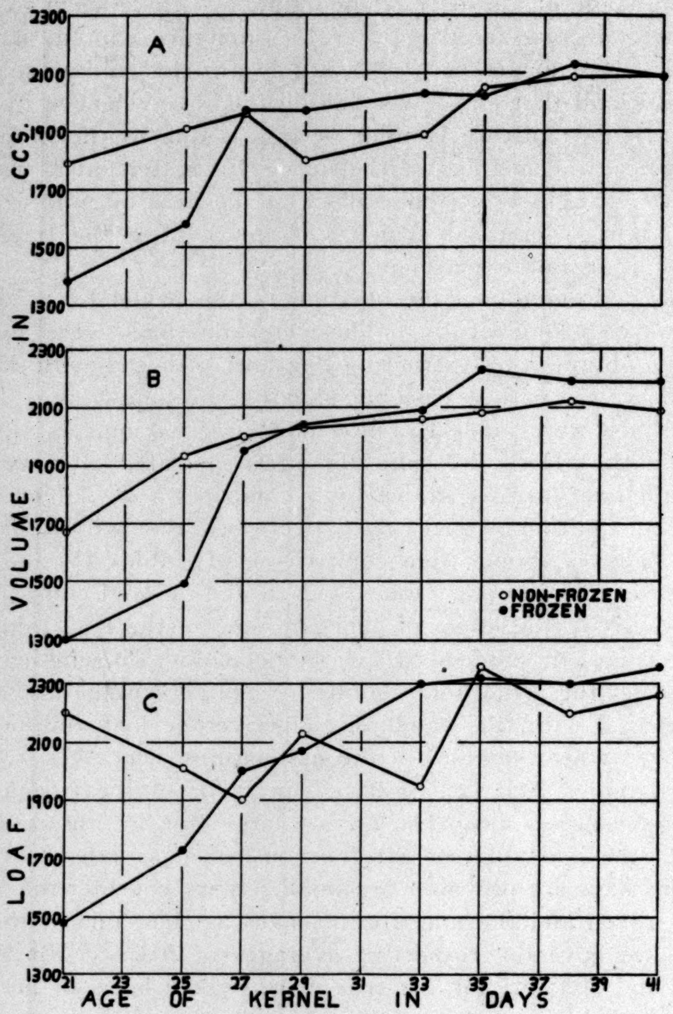

Fig. 1. Loaf Volume of Flour Milled from Frozen and Non-Frozen Wheat Harvested at Various Stages of Maturity

A. Milled Dec. 1923, baked Feb. 1924. Data from Table IV.

B. Milled Dec. 1923, baked Feb. 1924. Two cubic centimeters of normal lactic acid was added to each loaf at the time of mixture. Data from Table V.

C. Wheat aged 1 year, milled Dec. 1924, baked Jan. 1925. Data from Table VI.

and frozen samples respectively. The increase over the first baking is 101 cc. and 192 cc. respectively, and a percentage increase of —0.8 and 3.3 per cent respectively on the basis of the standard loaf. It we consider the standard loaf as unchanging, there is apparently an increase in loaf volume due to the addition of acid, and the increase due to aging of the wheat is not greater than the increase produced by the acid. On the other hand, the actual loaf volumes were greater at the time of the last baking. This indicates that there was a beneficial effect of aging which could not be accounted for on the basis of the addition of acid alone. Thus it is impossible to say, from these experiments, whether the beneficial effect of aging is due to the increase in acidity. The experiments, in a slight way, indicate the correctness of the previous findings of Whitcomb, Day, and Blish (1921)—that frosted wheat improves more on aging than does non-frosted wheat.

Wheat Frosted in the Field

As a basis for comparison between wheat frozen under the so-called artificial condition and wheat frozen in the field we have gathered together in Table VII the milling data on the wheat grown on plots of the Experiment Station in 1924. The wheat was planted at weekly intervals, so that some of the late plantings were subjected to frost. The baking data obtained with the flour milled from these wheats are given in Table VIII.

TABLE VII

Milling Data of Marquis Spring Wheat Planted on the Experimental Plots of the Station at Weekly Intervals, Beginning May 15, 1924
Wheat Milled Nov. 8, 1924.

Lab. No.	Wt. per Bu.	Total damage	Dark, hard, vitreous kernels	Protein in wheat (dry basis)	Dry crude gluten in flour	Flour yield	Ash in flour (dry basis)
	Lbs.	%	%	%	%	%	%
1833	60.2	0.9	58	14.57	11.7	73.8	0.60
1834	60.3	1.0	64	15.87	12.8	73.5	0.58
1835	60.7	2.3	63	15.93	13.2	73.9	0.65
1836	58.3	71.0	63	16.13	13.0	73.0	0.61
1837	56.5	91.5	90	15.89	12.8	69.5	0.70
1838	54.0	97.4	91	14.06	10.8	65.3	0.68
1839	47.6	100.0	85	14.55	7.8	69.1	0.89

TABLE VIII

Baking Data of Flour Whose Milling Data are Given in Table VII
The data are the average of bakings made Nov. 11, and Nov. 18, 1924.

Lab. No.	Absorption	Fermentation time	Loaf volume	Color	Texture
	%	Min.	cc.	%	%
1833	58	184	2110	96	98
1834	58	173	2095	96	98
1835	58	167	2010	95	96
1836	60	150	1895	94	95
1837	64	137	1830	88	87
1838	70	126	1600	70	63
1839	72	178	1230	30	40
Check	58	208	2205	100	100

The first three samples were not frosted, the last four were frosted. The damage, as indicated in Table VII, is frost damage. The first two samples gave nearly the same loaf volume while the frosted samples all showed lower loaf volumes, loaf volume decreasing as the sample became more immature. The question now arises—would samples grown under the same conditions and yet not frosted have given the same loaf volume if development had been stopped at the same stage at which the frost occurred. It is rather hard to draw conclusions from this series because of the abrupt change from practically no frost to 71 per cent frost. Another factor is operating here to make it impossible to draw valid conclusions as to the effect of frost. The frosted wheat was grown from later plantings than the non-frosted lots and consequently the frosted wheat was not grown under the same climatic conditions, for while the non-frosted samples were ripening in August the frosted ones were ripening in September and October. It is doubtful whether the differences in loaf volume of the non-frosted samples are greater than the experimental error, but it will be noted that loaf volume decreases with lateness of planting. Ripening progresses much more slowly in September than in August. It might justly be contended that the low loaf volume of sample 1836 and possibly of sample 1837 was not due to the effect of the frost but to other factors. In order to obtain the most reliable data from wheat frosted in the field, the wheat planted at the normal time should be frosted, and when such a frost occurs there is no non-frosted wheat grown under the same conditions for a standard of comparison.

These difficulties inherent in the study of the effect of frost on wheat frozen in the field led us to adopt the method of freezing the gathered heads artificially.

Discussion

It is not our intention to contend that freezing affects none of the properties of flour, for Shutt (1907), Blish (1920), and later, Sharp (1925) found that flour from frosted wheat contained more of the simpler nitrogenous compounds than did flour from non-frosted wheat; and it will be shown in later papers that other constituents of the wheat kernel are also affected by freezing. We interpret our results as indicating that the loaf volume is not affected by freezing alone if we use as our standard for comparison the loaf which the same wheat, non-frosted, at the same stage of development would give, provided the wheat contained less than about 46 per cent of moisture at time of freezing, and provided that freezing in the field does not produce effects which were absent in our method of experimentation. The effect of freezing in the field needs further investigation, especially in regard to its effect on the nitrogen compounds and carbohydrates.

Literature Cited

Birchard, F. J.
1920. Report of the Dominion Research Laboratory. Ottawa, Canada.

Blish, M. J.
1920. Effect of premature freezing on composition of wheat. J. Agr. Res. Vol. 19, pp. 181-188.

Sharp, P. F.
1925. Wheat and flour studies III. The amino nitrogen content of the immature wheat kernel and the effect of freezing. Cereal Chem. Vol. 2, pp. 12-38.

Shutt, F. T.
1907. Report of the chemist. Frosted wheat. Canada Expt. Farms Rept. pp. 140-143.

Whitcomb, W. O., Day, W. F., and Blish, M. J.
1921. Milling and Baking studies with wheat. Mont. Agr. Expt. Sta. Bull. 147.

Whitcomb, W. O. and Sharp, P. F.
1925. Germination of frozen and non-frozen wheat harvested at various stages of maturity. J. Agr. Res. Vol. 31, pp. 1179-1188.

Wilhoit, A. D.
1916. Annual report of the chief inspector of grain to the Minnesota Railway and Warehouse Commission. pp. 25-28.

FACTORS AFFECTING THE DIASTATIC ACTIVITY OF WHEAT FLOUR

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(Read at the Convention June 11, 1926)

The baking strength of flour is generally conceded to be due to two factors: (1) the gas-holding capacity of the dough and (2) the gas-producing capacity of the dough. The carbon-dioxide gas which extends the dough is produced by the action of yeast on soluble carbohydrates or sugar. Since the yeast requires a supply of soluble carbohydrates or sugar for gas production, the diastatic property, or sugar-producing power, bears an important relation to gas-producing capacity.

Rumsey (1922) devised a satisfactory method for determining diastatic activity of flour, and determined diastatic activity on samples of flour milled from wheat of different types and from different sources. Rumsey found considerable variation in diastatic properties of flours from different sources, and flours of satisfactory baking strength usually showed a relatively high diastatic activity.

Sherwood and Bailey (1926) found that it is possible to increase diastatic activity in flour by adding sprouted wheat, and the increased diastatic activity improved the baking quality of the flour.

The purpose of this study was to determine the factors affecting the diastatic properties of normal sound wheat, and their relative importance. The three principal factors studied have been (1) variety, (2) climate or rainfall, (3) soil fertility or cropping systems.

Experimental

For the present study samples of straight-grade flour were made. They were all prepared in a similar manner and milled on the small experimental mill. The wheat was sound, and free from weather damage.

The diastatic activity of the flour was determined by Rumsey's method, and results are expressed as milligrams of maltose formed from 10 grams of flour when digested 1 hour at exactly 27° C.

Variety

Different varieties of wheat are known to differ in baking quality, and studies of the diastatic properties of varieties were made in the hope that they would explain the difference in baking quality. For studies on variety, samples were selected from the variety plots of the Agronomy department at Fargo and the plots at the Dickinson and Williston substations, as the different varieties are produced under strictly comparative conditions.

Table I shows data on diastatic properties of different varieties from three locations in North Dakota for the 1923 crop. Table II shows similar data for two locations for the 1925 crop.

TABLE I

VARIATION IN DIASTATIC ACTIVITY OF WHEAT VARIETIES—1923 CROP
Data on straight-grade flour prepared on small mill.

Variety	Diastatic activity—mgm. maltose formed		
	Fargo plots	Dickinson plots	Williston plots
Marquis	57.4	90.1	75.0
Kota	103.5	164.5	114.3
Power Fife	83.9	111.6	88.2
Ruby	107.1	109.8	
Preston	85.7	119.7	
Turkey	.....	124.2	
Kubanka	160.1	240.6	221.4

It will be noted from Tables I and II that one variety, Kubanka durum, is significantly higher in diastatic activity than the others. Kota is intermediate between Kubanka and other varieties in diastatic activity. The other varieties listed show some variation, but the difference between them is not so striking or so significant as in the two cases mentioned. Marquis averaged lowest in 1923, but Ruby and Preston were lower than Marquis in 1925.

TABLE II

VARIATION IN DIASTATIC ACTIVITY OF WHEAT VARIETIES—1925 CROP
Data on straight-grade flour prepared on small mill.

Variety	Diastatic activity—mgm. maltose formed			
	Fargo plots Series 2*	Fargo plots Series 3†	Dickinson plots	Average
Marquis	104.2	86.5	66.5	85.7
Kota	132.4	113.7	134.6	126.9
Ruby	86.2	82.0	87.1	85.1
Preston	90.6	67.2	85.2	81.0
Ceres	85.5	84.9	101.6	90.7
Burbank Quality	84.9	74.5	85.2	81.5
Turkey Red	.....	....	82.6	82.6
Kubanka	164.4	....	224.5	194.5

*After red clover.
†After sweet clover.

Kubanka durum, as stated previously, shows distinctly high diastatic capacity, but does not give as good baking test as varieties which are lower in diastatic activity. The relatively low baking quality of Kubanka, as compared to Marquis, does not indicate that high diastatic activity is undesirable. The low baking quality of Kubanka is evidently due to the low gas-holding capacity of the dough. Kota has a relatively high diastatic activity and also a better gas-holding capacity than Kubanka, and under conditions in our laboratory usually gives a loaf of good volume.

Tables I and II show that considerable variation occurs within the same variety, owing to location and season.

Effect of Climate or Rainfall on Diastatic Activity

Bailey (1925) suggests that low diastatic activity may be associated with low rainfall or inadequate moisture supply. For the 1923 crop it was possible to make comparisons on wheat produced with and without irrigation at the Williston substation. Table III shows that irrigation increased diastatic properties of both Marquis and Kota wheats.

TABLE III

EFFECT OF IRRIGATION ON DIASTATIC PROPERTIES OF WHEAT GROWN AT WILLISTON—1923 CROP

	Diastatic activity—mgm. maltose formed	
Variety	Non-irrigated	Irrigated
Marquis	75.0	83.4
Kota	114.3	217.8

From the 1925 crop, samples of Marquis wheat from 11 different locations in the state were examined for diastatic properties. Table IV shows the diastatic activity and rainfall for these. The rainfall data are in all cases taken from the nearest Weather Bureau station. The correlation coefficient between total rainfall for May, June, and July and diastatic activity was 0.2941; and that between diastatic activity and July rainfall was 0.2651. The probable error in each case, owing to the small number of comparisons, was almost equal to the correlation coefficient, and these correlation coefficients cannot be considered significant.

It is probable, however, that the relation between rainfall and diastatic activity is in this case obscured by other factors. Soil type and soil condition are variables which may obscure the effect of rainfall unless the number of comparisons is sufficiently large to eliminate such factors.

The data in Table IV show that the same variety of wheat will vary in diastatic activity when produced under different conditions—and this variation may be due in part to rainfall variation, and in part to soil fertility or soil condition.

TABLE IV

RELATION OF RAINFALL TO VARIATION IN DIASTATIC ACTIVITY OF MARQUIS WHEAT—1925

Data on straight-grade flour prepared on small mill.

Where grown	Rainfall—1925 May	June	July	Total 3 Mo.	Diastatic activity mgm. maltose
Fargo	1.98	5.62	4.35	11.95	86.3*
Langdon	0.83	4.44	1.63	6.90	50.9
Edgely	0.55	8.03	2.26	10.84	93.7
Dickinson	0.89	4.31	1.29	6.49	67.8†
Dawson‡	1.52	4.68	0.42	6.62	67.5
Westhope	0.72	3.16	1.52	5.40	89.0
Drady§	0.57	3.56	1.00	5.13	79.8
Alkabo ‖	1.02	3.97	0.91	5.90	65.6
Crosby	1.40	3.92	0.72	6.04	72.2
Watford City	0.47	5.51	1.58	7.56	70.9
Valley City	0.67	6.72	0.98	8.37	96.9

*Average of three samples.
†Average of two samples.
‡Rainfall data from Steele, N. Dak.
§Rainfall data from Minot, N. Dak.
‖Rainfall data average of Howard and Crosby, N. Dak.

Effect of Soil Fertility and Cropping Systems

Table II gives results of diastatic activity on two series of variety samples produced at Fargo. In Series 2 the wheat was seeded on red clover ground, while in Series 3 the wheat followed sweet clover. Table II shows that without exception the diastatic activity is higher in Series 2 samples than in Series 3 samples.

For a more extensive study of the effect of cropping systems, samples of wheat were selected from the rotation and fertility plots of the Agronomy department, at Fargo. These plots were all seeded to Ceres wheat, a hybrid of Marquis and Kota.

The data on Ceres wheat are given in Tables V and VI. Since the soil type is in all cases the same, the only variable is the previous treatment the soil has received. The application of manure and phosphorus caused an increase in diastatic activity, but potash had a depressing effect.

TABLE V

EFFECT OF CROPPING SYSTEMS AND PRECEDING CROP ON DIASTATIC ACTIVITY

Data on flour from Ceres wheat from Fargo rotation plots, 1925.

Plot	Rotation	Diastatic activity mgm. maltose
2a	Continuous wheat	102.8
27a	Alfalfa 3 yrs, corn 2 yrs, wheat	99.4
5b	Corn, barley, millet, wheat	130.5
6b	Corn, barley, clover, wheat	145.4

The data presented show that the diastatic activity of wheat may be influenced by at least three variables—variety, rainfall or moisture supply, and soil treatment. Is difference in diastatic activity due to variation in the amount of enzyme present or to susceptibility of starch to diastase attack? The method of Rumsey measures the combined effect of both of these variables.

TABLE VI

EFFECT OF FERTILIZERS ON DIASTATIC ACTIVITY

Data on flour from Ceres wheat from Fargo rotation plots, 1925.

Plot	Fertilization	Diastatic activity mgm. maltose
5	Check plot	83.9
6	Fresh manure added	91.5
7	Fresh manure and phosphorus added	108.0
8	Fresh manure, phosphorus, and potash added	84.6
9	Fresh manure, phosphorus, potash, and lime added	118.5

Variation in Susceptibility of Starches to Diastatic Attack

Since Rumsey's method measures both the diastase concentration and the susceptibility of the starch or substrate to diastase attack, it was decided to eliminate one of these factors by using the same substrate. A cold-water extract was prepared of three samples of flour (Marquis, Kota, and Kubanka) showing wide variation in diastatic activity by Rumsey's method, and the clear solution obtained by decantation or centrifuging was used on a sample of commercial wheat starch.

Table VII shows results of substituting commercial starch for the flour as substrate. The three samples from the 1923 crop show almost the same diastatic activity when the same substrate is used. Of the 1925 samples, Marquis shows a lower diastatic activity with commercial starch than Kota or Kubanka, but the difference is considerably less than when flour is used as a substrate. The data in Table VII indicate that the concentration of diastase does not vary greatly in these six samples from the 1923 and 1925 crops. The variation in diastatic activity as obtained by Rumsey's method, in this case, therefore, is due largely to the variation in the susceptibility of starch to diastase attack.

TABLE VII

EFFECT OF SUBSTRATE ON DIASTATIC ACTIVITY

Wheat Varieties Grown at Dickinson in 1923 and 1925.

Substrate	Diastatic activity, 1923			mgm. maltose formed, 1925		
used	Marquis	Kota	Kubanka	Marquis	Kota	Kubanka
Flour of sample	90.1	164.5	240.6	66.5	134.6	224.5
Commercial wheat starch	50.4	48.7	44.4	43.6	62.3	60.9

Table VIII indicates that variation of diastatic activity in the same variety (Marquis) is also due to a large extent to the susceptibility of the starch to diastase attack.

TABLE VIII
EFFECT OF SUBSTRATE ON DIASTATIC ACTIVITY
Marquis wheat grown at four different points in No. Dak., 1925 Crop.

	mgm. maltrose formed			
Substrate used	Fargo	Dickinson	Epworth	Crosby
Flour of sample	104.2	66.5	99.1	72.2
Commercial wheat starch	44.2	43.6	46.4	53.4

In the experiments just cited, the substrate was kept constant and the diastatic preparation varied. Table IX shows the result of using the same diastatic preparation on different samples of starch. Three samples of starch were used for this experiment: (1) A, the commercial wheat starch used previously; (2) B, a sample of starch prepared in the laboratory from a strong hard spring patent flour; and (3) C, a sample of starch prepared in the laboratory from a durum wheat patent. A cold-water extract of Marquis flour was used as a source of diastase. The durum wheat starch shows a significantly greater sugar production than either of the other samples.

TABLE IX
EFFECT OF SUBSTRATE ON DIASTATIC ACTIVITY
Diastatic extract from flour from Marquis wheat grown at Fargo, 1925.

	mgm. maltose formed			
Substrate used	Flour of sample	Starch A	Starch B	Starch C
Diastatic activity	104.2	44.2	10.1	182.0

The diastatic activity of flour as measured by Rumsey's method is the result of the combined effect of diastase concentration and the relative susceptibility of the starch of the flour to diastase attack. The durum wheat starch is apparently much more readily attacked by diastase than starch from such hard red spring varieties as Marquis.

Discussion

The data presented are the result of preliminary investigations and show that the diastatic activity of wheat is affected by at least three factors. Variation in diastatic properties may be due to the nature of the substrate present as well as to the concentration of diastase. In case of varietal and other variation, the concentration of diastase appears to be relatively unimportant, and the difference in diastatic properties is evidently due to the nature or structure of the starch granules.

The diastatic activity of spring wheats, from the data secured, appears to be relatively low, and low diastatic activity may often be a limiting factor in determining baking quality. Since variation in diastatic activity is due in part to the nature and condition of the starch present, the possibility is suggested of modifying the diastatic properties of wheat during the milling process.

Summary

1. Kubanka durum showed distinctly higher diastatic properties than other wheats examined, and Kota wheat was intermediate between Kubanka and the other spring varieties.

2. Marquis wheat produced at different points in the state showed variation in diastatic properties, and data indicated that low diastatic activity may be associated with low rainfall.

3. Ceres wheat produced on rotation and fertility plots at Fargo showed variation in diastatic activity due to different cropping systems and fertilizers added.

4. The variation in diastatic activity of flour appears to be due in large part to the susceptibility of the starch granule to diastase attack, rather than to the concentration of diastase present.

Literature Cited

Bailey, C. H.
1925. The chemistry of wheat flour. Chemical Catalog Co., New York.

Rumsey, L. A.
1922. The diastatic enzymes of wheat flour and their relation to flour strength. Am. Inst. Baking, Bull. 8.

Sherwood, R. C. and Bailey, C. H.
1926. Control of diastatic activity in wheat flour. Cereal Chem. Vol. III, pp. 107-136; 163-182.

THE DETERMINATION OF MOISTURE IN FLOUR
A REVIEW OF RECENT WORK

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Introduction

The determination of moisture in cereal products is a familiar activity in the daily laboratory routine of flour mills and bakeries. The chemist is responsible for trustworthy results, as moisture is an essential factor in quality of product and manufacturing costs. Wheat flour is hygroscopic, and moisture content has highly important relations to standard weight laws; and its influence on spoilage is a problem of considerable significance to the miller and baker.

Limits for the moisture content of flour and bread have been established in connection with definitions and standards by the United States Department of Agriculture for the guidance of officials charged with the enforcement of the Food and Drugs Act.

An equitable and satisfactory method for the determination of moisture is fundamental to the proper interpretation of these definitions and standards because of their important economic and legal aspects.

The study and development of methods of analysis that may eventually come before the courts in cases brought under the Food and Drugs Act is a subject in which every cereal chemist has an immediate interest. The Association of Official Agricultural Chemists, which is responsible for the adoption of such methods, has followed a liberal policy of encouraging co-operation with industrial chemists and others who are directly interested. The American Association of Cereal Chemists has recently begun to appreciate the value of co-operation with the referees of the above organization in collaborative studies of methods for the analysis and examination of cereal products; and closer relations between the two associations have been productive of mutual benefit.

Recently considerable attention has been devoted by the A. O. A. C. to a critical study of the methods proposed for the determination of moisture in wheat flour, and co-operative studies have also been started on the determination of moisture in bread.

The moisture determination has also been the subject of study by others, so that a rather considerable literature has been accu-

mulated in the last twenty years since the fixing of a moisture limit of 13.5% for flour. It is the purpose of this discussion to review some of the more important recent contributions on the determination of moisture in wheat flour.

Discussion

The inherent difficulties in the way of making an accurate determination of the total water or moisture content of foods composed largely of colloids is well recognized by investigators.

Nelson and Hulett (1920), in a study of the moisture content of cereals supplementing previous work by the latter on the moisture content of coal, have emphasized some of these difficulties. "The layer of water absorbed on the surface of colloids has quite different properties from ordinary water. Its vapor pressure is much lower and if this layer is of molecular dimensions it cannot be removed by the best dessicating agents in a vacuum desiccator." Colloids will not part with all their water when subjected to air-drying temperatures from 100° to 110° C., or to exposure over dehydrating agents at ordinary temperatures and pressures. The vapor pressure continually decreases with the removal of water until the system reaches such a low vapor pressure that water is no longer obtained, altho considerable may be present.

Wheat flour is composed of relatively small aggregates of the grain or kernel tissues, which are complex systems built up largely of organic compounds, proteins, carbohydrates, and fat. Organic compounds when subjected to heat, decompose with the production of gases, CO_2, CO, CH_4, H_2, moisture, and a residue. There is probably no definite temperature of decomposition, but reactions proceed at all temperatures at different rates. "A substance that decomposes readily at 200° to 300° C. also decomposes at lower temperatures, but often so slowly that no measurable results are obtained in experimental time." Nelson and Hulett, by the use of an ingenious apparatus for the maintenance of a very high vacuum, the measurement of gas produced by thermal decomposition, and the weighing of the condensed water, were able to show that when wheat flour is heated to 184° C. for 4 hours, only 0.4 cc. of gas per gram of sample was observed. The graphs of Figure 3, of their paper, indicate that there is no break in the curve for moisture percentage until 184° C. is reached. A sample of wheat flour protein also showed a deviation in the curve at this temperature.

The work of Nelson and Hulett is most important in providing means for determining the temperature and the duration of time to which an organic substance like wheat flour may be exposed without showing water and gases of decomposition.

In view of this work it is probable that the so-called water of hydration or constitution may not be so important relatively as some investigators have supposed it to be, altho it is useless to speculate on the distribution of water in wheat flour on the basis of the amount held by surface phenomena (adsorption); water of hydration, or constitution of proteins and carbohydrates; and water of crystallization, until this problem has been further attacked by physical chemists.

At the present stage in the study of the moisture determinations, it is generally agreed that the problem is to establish a method which will express moisture percentage in weight of a flour sample (obtained from some accurate method of sampling) by drying under standardized conditions clearly defined on the basis of comparative and co-operative work.

It is well known that there has been a wide variation in the methods used by chemists for this determination, and naturally a considerable amount of investigation has been undertaken by various organizations in an attempt to survey existing laboratory practices.

The American Institute of Baking (1921) was able to secure the co-operation of 28 laboratories, including those of flour mills, bakeries, state experiment stations, consulting chemists, and others engaged in control or commercial work, who determined the moisture on three samples of flour sent to them by the Institute with the recommendation that they report the method used. The results of this survey showed striking variations in the methods and equipment employed and of course rather wide variations in the percentages of moisture reported.

At this time the official method of the A.O.A.C. was based on drying in hydrogen or in vacuo at the temperature of boiling water to constant weight (approximately five hours). It is worthy of comment here that the reports from but 8 laboratories might be construed as following the official methods, and some of these were using higher or lower temperatures than that of boiling water, while but one analyst reported drying in hydrogen. Seven laboratories reported the use of vacuum ovens, but one of these dried one hour at 215-220° F. Eighteen were equipped with air ovens, mainly of the electrically heated type, but some used water and toluol

walled ovens. The temperature of air drying ranged from 100-105° C. Two reported the use of vacuum desiccators at room temperature, and in one case a total drying period of 60 hours. A closer agreement was reported in the results obtained from the vacuum methods than with the air ovens. As a matter of interest in passing, we will cite some of the results obtained from the use of water ovens and vacuum ovens, also a toluol oven at 105° C.

TABLE I
PERCENTAGE OF MOISTURE AS DETERMINED IN DIFFERENT TYPES OF OVENS

Samples of flour	A	B	C
	%	%	%
Analyst 6 Water oven 100° C.	12.00	10.48	12.45
Analyst 11 Water oven Temp. ?	10.70	9.03	11.21
Analyst 28 Toluol oven 105° C.	11.95	10.35	12.61
Analyst 2 Vac. oven at 100° C.	12.45*	10.87*	13.16*
Analyst 3 Vac. oven Temp. B. W.	12.35*	11.00*	13.00*

*Av. 2 determinations.

This survey, like others, indicated the necessity for adhering to some standard procedure, leaving aside any question of "personal coefficient" or technic which may be involved, and it is somewhat suggestive that in spite of the existence of official methods, the A.O.A.C. method, and that recommended by the American Association of Cereal Chemists which dried flour in an air oven for 5 or 6 hours at 103 to 105° C., there was not more uniformity shown in the choice of a method.

The present tendency in the study of methods for the determination of moisture in flour has been to eliminate, as the result of critical study, a number of procedures that were at one time considered applicable. Among these is the use of dehydrating agents in vacuum desiccators in which flour is exposed until it no longer loses weight. Some of these investigations have been published in the Journal of the Association of Official Agricultural Chemists as reported by various referees, White (1915), McGee (1916), LeClerc (1920), and the last has stated that the vacuum desiccator method. using calcium oxide, gave results as good as those secured by the official method. The results of such methods, however, show marked variations in the amounts of moisture obtained, and they are in general time-consuming and subject to criticism not only on the grounds referred to by Nelson and Hulett (1920), but because of the difficulty of securing agreement on initial pressure, and fluctuations in the composition of the dehydrating

agent, which require careful attention to obtain trustworthy results. The removal of water by reaction with calcium carbide and measuring the acetylene formed equivalent to the water present, after McNeil (1912), does not appear to have been extensively studied. The direct determination of moisture as proposed by Bidwell and Sterling (1925), based on the distillation of water from the material in the presence of toluene, is of considerable interest and the results reported by them on flour show close agreement with the vacuum method. It possesses several advantages in respect to the time consumed (less than an hour) and in the direct determination of the water.

An extensive series of four papers on the determination of moisture in wheat flour has been published by Snyder and Sullivan (1924, 1925, 1926) which presents a critical study of the methods used by the analyst:

1. Drying for 5 hours or longer in a water oven heated to the temperature of boiling water.

2. Drying for 5 hours or longer in air-heated ovens at 100° C. or higher.

3. Distillation methods. Brown-Duvel for grain.

4. Drying in vacuum ovens with the use of wide ranges in temperature and vacuo.

5. Vacuum drying without heat.

6. Drying in hydrogen and other "inert" gases to prevent oxidation.

Three methods were discussed in their first paper—drying in water-heated ovens, in air-heated ovens, and in vacuum ovens.

According to their Table I, the average difference in moisture percentage between drying in a water oven at the temperature of boiling water and in an air oven at 105° C. for a definite 5-hour period on 28 samples of flour was 1.43%, maximum difference 1.90%, minimum difference 0.82%.

These authors contend that "the main portion of the free moisture of flour is given off in water-oven drying. Much of that expelled at higher temperatures is in a different form." They ap pear to believe that after the limit of water-oven drying is reached, water is held "as physically bound," and another portion is held in chemical combination as water of hydration of proteins and carbohydrates. They recognize, however, the difficulty of quantitatively partitioning the water in flour.

Comparative tests are reported between the water-oven and the vacuum method, the latter at various pressures from 500 to 740 mm., noting temperature difference, time of evacuation, and time of heating. The average difference between the two methods was reported as 1.87%. Twenty-seven samples were tested. As wide a difference was reported as 2.32%. The vacuum method is criticized on the grounds of fluctuation in pressure and inequality in temperature in the chamber.

Drying in a water oven at the temperature of boiling water is also a variable term, as the boiling point varies according to the altitude of the locality.

Snyder and Sullivan contend that the vacuum oven is essential in flour investigations when it is necessary to determine approximately the loss under maximum conditions, but that its use exceeds water-oven drying from 1-3% because of the "different forms in which water is present in flour."

Their second paper (1924) is concerned with drying flour over desiccating agents, in order to determine if a method could be obtained which would give only the "free moisture" of flour without including other forms or causing chemical changes. They were unable to obtain satisfactory results with desiccating agents, sulfuric acid, or phosphorus anhydrid.

They also note that when flour is dried above the boiling point of water, chemical and physical changes occur; starch for example, is changed at very low temperatures.

The third contribution of these authors (1925) is concerned with the Brown-Duvel test for moisture in wheat and a comparison of the double-walled-flask method with the water-oven and vacuo drying.

The so-called double-walled-flask method is a distillation method with oil at 190° C. Nine tests gave an average of 12.3% moisture against 11.78% in water-oven drying. Further comparative results between water-oven and vacuum-oven drying were reported. The vacuum was 400 mm., time 5 hours, temperature 100° C. Nine determinations showed an average difference of 1.94%. The need for a rapid method for commercial work is suggested.

"It would seem feasible to determine the moisture as total volatile products by drying flour in a strictly empirical way, heating to a higher temperature than the boiling point of water, as 125°-135° C. for a short period, as one hour, and then applying

an established factor for correcting the results to conform to the water-oven basis of the standard official methods as specified."

Higher results are obtained by such a method than by the standard Brown-Duvel method for wheat or flour. As previously stated, such a procedure is in harmony with moisture tests under the provisions of the United States Grain Standards Act.

Snyder and Sullivan (1926) have recently published a fourth paper in their series of critical studies on drying flour in an atmosphere of inert gas. They used a modified form of Winton's apparatus, originally developed at the Connecticut Agricultural Experiment Station for drying in hydrogen, and reported percentages of moisture obtained with this apparatus, temperature 98.5° C. inside drying tubes; and the vacuum method at 98.5-101° C. for 5 hours at a pressure stated to be 600-738 mm. An average of 58 tests gave moisture percentage of 13.46% by the vacuum method, and 13.95% by drying in hydrogen.

They also made tests in which nitrogen was used as the inert gas. The results obtained showed an average difference in moisture percentage between this method and the vacuum of 0.434%, somewhat lower than the difference obtained with hydrogen as reported in their Table 2, which was 0.52%.

Observations were also made using a current of air, unheated and heated. It was found that air could not be dried satisfactorily without preheating. A comparison of drying in vacuum-oven heated air, the hydrogen is recorded in their Table 2, which indicates that the average difference in moisture percentage between drying in hydrogen and in air was 0.111%.

This observation seems to be in harmony with the work of Shutt and Moloney (1917)—that there is no appreciable oxidation of flour by drying in air.

According to Sullivan and Snyder satisfactory "drying of flour in tubes placed within copper tubes heated to the temperature of boiling water depends mainly upon (1) a uniform flow of perfectly dry gas or air, and (2) the loading of the tubes in such a way that the drying medium readily passes over and through the flour." The method is stated to be unsuitable for routine work, but satisfactory as a research method. Hydrogen and other gas drying is stated to represent more nearly the total moisture content of flour than other methods operated at the same temperature.

The authors conclude by defining the term "total moisture" or "water in flour" as "that which is held in loose and firm physical combinations with the flour particles, also that held in condi-

tion closely approaching chemical combination with the starch micellae, or water of hydration of protein." They also state that as an "industrial problem the determination of moisture in flour for manufacturing or other control purposes is largely a matter of using and strictly adhering to empirical conditions as to temperature, time, and manipulation.

"Any moisture method thus far developed is a relative rather than an absolute expression as to moisture content."

In line with the present tendency to study the various controllable factors in drying methods, Mitchell and Alfend (1924) have shown that when flour is dried in vacuo in covered dishes, results are invariably higher and check more closely than those obtained in open dishes, 0.60% higher on the average. When open dishes are used it appears to be immaterial whether the covers are placed under the dishes or removed from the oven. The difference in the results obtained from the covered and the open dishes is presumably dependent upon the time of exposure of the dishes to air before they were covered. It required 15 minutes to release the vacuum with safety and it was impossible with their apparatus to eliminate time of exposure of the dried samples to the air. It required, in addition, 10 minutes when a large number of samples were run to cover the dishes and transfer to the desiccator.

An important contribution to the quantitative determination of moisture in wheat flour has been made by Spencer (1925). This painstaking investigation was conducted along the following lines:

1. A trial of methods that have hitherto been used at the Bureau of Chemistry for the drying of flour.

2. A comparison of results obtained by drying at reduced pressures.

3. The development of a vacuum method to be used as an umpire method.

4. The development of a rapid method to be used as a routine method. Conditions and equipment common to the experimental work—as weight of flour, containers (covered), vacuum oven, electric oven, desiccating agents, vacuum pump, partial pressure readings, and temperatures—were specified and described. Observations on the humidity of the days when the samples were weighed were also noted.

Two methods for drying in hydrogen were described and used, but the results obtained were irregular and unsatisfactory.

This is contrary to the later experience of Snyder and Sullivan (1926) who were able to establish satisfactory conditions as noted above.

Graphs 1 to 4, published by Spencer, show the percentages of moisture obtained from three different types of flour on different days by (1) the water-oven method, temperature 98-100° C., time, 5 hours, dishes uncovered; (2) vacuum-oven method, 127-203 mm. pressure, temperature 98-100° C., time, 5 hours, dishes uncovered; (3) vacuum method at 70°, pressure 127-203 mm. time 5 hours, dishes uncovered; and (4) electric oven, temperature 111° C., time 5 hours, dishes uncovered. Humidity observations on the days the samples were weighed are also graphed. The average results are recorded in Table II.

TABLE II
PERCENTAGE OF MOISTURE REPORTED BY SPENCER WHEN FLOURS WERE DRIED UNDER DIFFERENT CONDITIONS

Flour	Water oven	Vac. oven 98-100° C.	Vac. oven 70° C.	Electric oven 110° C.
	%	%	%	%
Hard Spring patent	11.61	13.01	11.60	12.49
Hard Spring clear	9.92	11.33	10.01	10.88
Soft wheat straight	12.48	13.82	12.51	13.33

The results reported showed fairly close agreement on any one day, but were rather irregular on different days, when the sample was weighed out under different atmospheric humidities.

The data from these four methods were obtained with uncovered dishes, but a series of results on drying in vacuo at 98-100° C. at 25 mm. pressure for 5 hours, using in one series covered dishes and in the other loosely covered dishes, was published which indicated in general the tendency toward higher results when the dishes were covered, as suggested in the work cited above by Mitchell and Alfend (1924).

Spencer has proposed a standard vacuum method which is formulated in detail and a routine method based on drying in air at 130° C. for 1 hour, in which conditions are also defined, but more suitable for commercial work, and results are presented indicating a close agreement between these two methods.

Co-operative studies conducted by Spencer as associate referee on cereal products of the Association of Official Agricultural Chemists have corroborated this experience. The methods of vacuum drying and air drying at a relatively high temperature and short exposure are still being studied by the American Association of Official Agricultural Chemists.

Smith and Mitchell (1925) have extended the work of Mitchell and Alfend (1924) to an important study of the comparative results of drying flour in the water-jacketed vacuum oven, the water oven, and the electric oven, the variation that is obtained in check determination of moisture when the samples are placed in different positions in the same oven, the losses or gains in weight of flour exposed to the air before and after drying, and the effect of cooling the sample under different conditions before the final weighing.

The highest moisture percentages were obtained by the vacuum oven, the loosely covered dishes giving higher and more uniform results than the open dishes. Flour dried at atmospheric pressure in loosely covered dishes gave variable results.

When the flour was dried on two different days, in different positions in the same ovens, vacuum (7) water (2) and electric (3) the vacuum oven gave higher and more uniform results than the electric oven and decidedly more uniform results than the water oven as indicated in their Table II.

"Duplicate determinations showed as high a variation as 0.47% while check duplicate determinations gave as wide a variation as 0.55%, even though all the samples both for duplicate and check duplicate determinations, were weighed at the same time and dried under conditions as nearly identical as possible with the exception of the position of the sample in the oven."

The necessity for taking special precautions to insure that the samples of flour will not be unduly exposed to the atmosphere during weighing and after removal from the oven during cooling is also shown by Smith and Mitchell, and in addition the inefficiency of calcium chloride as a desiccator dehydrating agent before making the final weight.

Shuey (1925) has also studied the vacuum method and the "routine" method as proposed by Spencer in connection with collaborative studies on moisture made by the American Association of Cereal Chemists, but also in one series of tests decreased the temperature of heating to 125° C. and in another series increased it to 135° C. but maintained the period of exposure to heat at 1 hour.

Shuey's interpretation of the results obtained states that the routine method is dependable within these ranges of temperature, that the four methods are fairly concordant, and recommends further study of the "routine" method for the mill laboratory.

It is interesting to note in this connection that such rapid methods employing high temperatures and short periods of exposure to heat at atmospheric pressures have been used in Europe for the determination of moisture for some time. Among them is the apparatus of Meihuzen, of Veenham, Holland, who found that drying wheat flour 4 minutes per gram at 180° C. gave good results.

It is very probable, according to the work of Nelson and Hulett previously cited, that at a temperature of 100° C. or about that, the moisture cannot be removed from colloidal organic matter like flour, and a higher temperature is required. It is doubtful whether there is not some adsorbed water present even at temperatures when decomposition becomes rapid. These investigators found little increase in water due to decomposition at 180-184° C., but a considerable percentage of moisture over that found at 100-100° C.

The use of a higher temperature at which nearly all the moisture is removed appears to give more concordant results. A method such as the proposed "routine" method with a relatively high temperature is a development in this direction, and if strictly carried out according to instructions should point the way to rapid and satisfactory moisture determinations with a minimum of time and equipment.

These and other critical and related studies of methods for the determination of moisture in flour have brought about a more general appreciation of the necessity for employing a standard procedure which is as rigidly controlled as it is possible to make it, based on the critical study of collaborative work undertaken by those interested.

Literature Cited

American Institute of Baking.
1921. Methods of analysis, co-operative work, cereal products, wheat flour. Bull 2, pp. 4-35.

Bailey, C. H.
1920. The hygroscopic moisture of flour exposed to atmospheres of different relative humidity. J. Ind. Eng. Chem. Vol. 12, pp. 1102-1104.

Bailey, C. H.
1921. Report on cereal foods. J. Assoc. Off. Agr. Chem. Vol. 5, pp. 241-245.

Bidwell, G. L., and Sterling, W. F.
1925. Preliminary notes on the direct determination of moisture. J. Assoc. Off. Agr. Chem. Vol. 8, pp. 295-301. J. Ind. Eng. Chem. Vol. 17, pp. 147-153.

Le Clerc, J. A.
1920. Report on cereal foods. J. Assoc. Off. Agr. Chem. Vol. 3, pp. 447-452.

McGee, W. J.
1916. Water in feeds and feedingstuffs. J. Assoc. Off. Agr. Chem. Vol. 2, pp. 120-123.

Mangels, C. E.
1924. Report on cereal foods. J. Assoc. Off. Agr. Chem. Vol. 8, pp. 140-149.

Mitchell, L. C. and Alfend, S.
1924. The determination of moisture in flour. J. Assoc. Off. Agr. Chem. Vol. 8, pp. 76-79.

Nelson, O. A. and Hulett, G. A.
1920. The moisture content of cereals. J. Ind. Eng. Chem. Vol. 12, pp. 40-52.

Shutt, F. T. and Maloney, P. J.
1917. The determination of the moisture content of flour. Trans. Roy. Soc. Canada, Vol. 11, Series 3, pp. 101-105.

Snyder H. and Sullivan, B.
1924, 1925, 1926. Determination of moisture in wheat and flour. J. Ind. Eng. Chem. Vol. 16, pp. 741-744; Vol. 16, pp. 1163-1167; Vol. 17, pp. 311-314; Vol. 18, pp. 272-275.

Snyder, H.
1922, 1923. Wheat flour, its weight and moisture content. pp. 55, N. D. Published by the Millers National Federation, Chicago, Ill.

Shuey, G. A.
1925. Collaborative study of moisture methods. Cereal Chem. Vol. II, pp. 318-323.

Spencer, G. C.
1925. The quantitative determination of moisture in wheat flour. J. Assoc. Off. Agr. Chem. Vol. 8, pp. 301-311.

Smith, E. R. and Mitchell, L. C.
1925. Observations on the drying of flour with notes on its hygroscopic properties. J. Ind. Eng. Chem. Vol. 17, pp. 180-182.

LEAVENING AGENTS FOR SELF-RISING FLOUR

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(Read at the Convention June 8, 1926)

While many millers may have felt that there is not the demand for their product they would enjoy having, the full significance of this fact was probably not realized until the recent presentation of statistics by Anderson (1926) showing that the present per capita consumption of flour in our country is approximately 25% less than it was 50 years ago. While it is freely admitted that the substitution of other foods, made possible by our high standard of living, is chiefly responsible for this change, it is conceded that those who bake their own bread use more flour, both in toto and per capita, than those who buy their bread baked. Whatever, within reason, can be done to retain this larger demand should, in itself be justification. Many millers, particularly of soft wheat, realizing the trend of the times, have sought to make home baking more agreeable to the housewife by supplying ready-mixed ingredients in self-rising flour. This work was undertaken mainly with a view of helping to determine accurately what formula for each particular flour would give the best results.

A great deal has been done in determining the factors affecting yeast-levened bread, but very little by comparison in studying the factors affecting chemically leavened bread. Jacobs (1922) criticized the lack of uniformity in the proportions of leavening agents and the poor quality of some self-rising flours. Bailey (1923), after examining about 90 samples, concluded that "a large proportion of the self-rising flours taken from the ordinary channels of trade was capable of producing biscuits that had good color and appearance. They were well leavened and palatable." Moher (1923) pointed out some of the problems of self-rising flour and suggested further investigation of certain features. Smith and Bailey (1923) studied the effect of the residual salts of chemical leavening agents on the properties of bread. Chittick and Dunlap (1925) proposed a method of measuring biscuit volume.

To correlate this work and build upon it an acceptable method of judging the quality of chemically leavened bread is the purpose of our present endeavor. Biscuits are chosen as the most representative bread using chemical leavening agents, and a score card is arranged for judging their quality. In order to compensate for the personal factor and the variation in weather and oven

conditions, a standard is baked each day using a high grade 40% patent soft wheat flour, properly leavened, to which the biscuits to be tested are referred for a comparison of each characteristic. In this way a total score is obtained which is comparable from day to day and may be used to reflect in a single figure the relative quality of different self-rising flours even tho baked at different times.

BAKING TEST

A typical formula in making tests is:

Flour	200.0 grams
Shortening	30.0 "
Water	114.0 "
Phosphate	3.75 "
Soda	3.0 "
Salt	3.5 "
Time of mixing	4 min.
Time of kneading and cutting	2 "
Time of baking	15 "
Height of roll	3/8 inch
Temperature of oven	475-500° F.

Each biscuit is surrounded by a brass ring and covered with a tin cover.

Each ring is ....1.9218 inches in diameter
1.5625 " " height.

The dry materials are sifted together four times. The solid shortening is cut in with a fork and the water added. The kneaded dough is placed inside an embroidery hoop three eighths of an inch high on a tin plate and rolled to an even thickness, the excess dough being pressed over the sides of the hoop. For determining volume and lightness, biscuits are cut from the dough in the hoop with 2-inch brass cylinders, following the method of Chittick and Dunlap (1925), which consists in baking the biscuits in uniform brass cylinders and measuring the volume of the biscuit by filling the unoccupied space in the rings with rape seed, thus using the cylinders themselves as the measuring device. All calculations are based upon weights, and results can be duplicated within a maximum variation of ±1.0%.

An improvement in accuracy is made on the original method by feeding the seed from a stoppered funnel located at a fixed height of 1 inch above the rings. Besides the specific volume (cc. of biscuit per gram of biscuit) proposed by Chittick and Dunlap (1925), which we have used as a measurement of "lightness," we weigh the unused dough and determine by proportion the flour actually used in the biscuit. This allows a volume measurement to be made based on the flour (cc. of biscuit per gram of flour). This appears in the score card as "Volume." Volume and lightness

are determined from the biscuits baked in the cylinders, and the other characteristics are judged from biscuits baked in open pans in the usual way.

Score Card

Despite the disadvantages of an arbitrary score card, it seems essential that the various characteristics which go to make up the total bread quality should be judged on the basis of their proportional value. It is considered necessary to give first place to flavor (30 points out of a total of 100) for it is a primary requisite that a food product have a pleasing taste and odor. Unfortunately, however, there is probably no other characteristic so subject to individual discrimination and personal error. Texture is considered next in importance, and to it is assigned a total value of 20 points, divided as follows:

Grain-size and uniformity of cells	5
Tenderness	10
Flakiness	5

Volume is valued at 15, and lightness at 10, and the other qualities in lesser values as shown in the typical score card in Table I.

TABLE I
SCORE CARD FOR BISCUITS
Subject: To Determine the Best Self-Rising Formula. Flour: Soft Wheat Patent.

		Score	A	B	C	D	E
Phosphate			3.50	3.75	4.00	4.25	4.50
Soda			3.00	3.00	3.00	3.00	3.00
Salt			3.50	3.50	3.50	3.50	3.50
pH of biscuit			7.8	7.5	7.2	7.0	6.8
1. General appearance		10					
(a) Color	5		4.25	4.35	4.50	4.55	4.60
(b) Size and uniformity	5		3.70	3.90	4.00	4.00	4.00
2. Crust		5					
(a) Tenderness	3		2.35	2.40	2.50	2.52	2.50
(b) Thickness	2		1.25	1.30	1.35	1.35	1.20
3. Texture		20					
(a) Grain, size and uniformity of cells	5		3.90	4.50	4.90	4.80	4.80
(b) Tenderness	10		8.00	8.75	9.00	8.75	8.50
(c) Flakiness (allows layers to be pulled off)	5		4.50	5.00	5.00	5.00	4.50
4. Flavor		30					
(a) Taste	20		17.00	19.00	20.00	19.50	18.00
(b) Odor	10		9.00	9.50	10.00	9.00	8.00
5. Crumb color		10	8.50	8.80	9.00	9.10	9.40
6. Lightness		10	8.74	9.17	7.96	7.60	7.46
7. Volume		15	12.47	12.67	12.10	11.80	11.50
Total		100	83.66	89.34	90.31	87.97	84.46

Conditions

Flour	200 gm.
Shortening	30 gm.
Water	108 cc.
Temp.	475-500° F.
Time	15 min.
Barometer	756 mm.

Effect of Water and Kneading

The effect of varying proportions of water is shown in Table II and the effect of kneading in Table III. As soon after adding the water to the mixed flours as the dough can be handled, it is picked up and folded by hand with the effect that the biscuit quality is increased up to a certain number of folds beyond which point the biscuits become tougher and of poorer grain.

TABLE II

EFFECT OF VARYING PROPORTIONS OF WATER

Water per 200 gm. flour cc.	Biscuit score %
100	85.50
104	86.30
108	86.87
110	87.24
114	87.75
118	84.35
122	81.66

TABLE III

EFFECT OF KNEADING DOUGH

No. of folds cc.	Biscuit score %
A. Folded with spatula to hold together	83.96
B. A + 4 folds	84.28
C. A + 10 folds	86.25
D. A + 15 folds	86.11
E. A + 20 folds	85.14

pH of Finished Biscuit

The pH of the finished biscuit is a valuable help in indicating correct proportion of leavening agents. In a series of more than five hundred bakings made from more than twenty-five different flours ranging in kind from Kansas hard wheat to Ohio soft wheat, the best biscuit quality was obtained when the pH range was 7.0-7.3. We find that this can be easily gaged by striping the opened biscuit with standard indicator solution of phenol-sulphone-phthalein (phenol red) the result checking very closely that obtained with the extraction method described by Smith and Bailey (1923). It is necessary, therefore, in order to secure the optimum result, to regulate the proportions of the leavening agents to secure this pH (7.0-7.3) in the baked product.

Proportions of Leavening Agents

While baking powders are made by combining one or two of a variety of acid agents with bicarbonate of soda, mono calcium phosphate is the only acid salt used commercially in self-rising flour. The determination of the neutralizing value of mono cal-

cium phosphate has been variously discussed in the last few years. The principal method in commercial use is that described by Warning (1923) and Adler and Barber (1925), which consists in completely neutralizing the mono calcium phosphate with excess caustic soda and back-titrating with acid. While this method is practical for comparing different phosphates, it is obvious, since it takes no account of the differences in flours themselves, that it cannot be relied upon in every case to indicate the correct proportion of phosphate and soda for each self-rising flour. Using mono calcium phosphate having a neutralizing value of 82.7 according to the above test, we have found it necessary, in order to secure the optimum baking results with different flours, to vary the proportions of phosphate and soda to such an extent that indicated neutralizing values were obtained ranging from 70 to 100. We recommend, therefore, that the mill chemist, in the manufacture of self-rising flour, determine by comparative baking tests the proportion of leavening agents best suited to each flour. Having found the proportion of phosphate and soda which will give a pH 7.0-7.3 in the baked biscuit, the quantity of soda which will give maximum quality should next be determined, varying, of course, the phosphate with the soda.

Conclusions

1. A baking test and score card based upon a standard are proposed for measuring the biscuit quality of self-rising flours.
2. There is an optimum quantity of water which may be added and an optimum amount of kneading for biscuit doughs.
3. The pH of the finished biscuit is a valuable aid in determining the proper proportion of leavening agents.
4. Flours differ in the quantities of acid and alkaline leavening agents required.

Literature Cited

Adler, H. and Barber, G. D.
1925. A study of the determination of the neutralizing value of mono calcium phosphate. Cereal Chem. Vol. II, pp. 389-390.

Anderson, Sydney
1926. Has the per capita consumption of wheat flour in the United States declined? Millers' Nat'l Federation Bull. 447.

Bailey, L. H.
1923. An examination of some self-rising flours. J. Am. Assoc. Cereal Chem. Vol. 8, pp. 119-124.

Chittick, J. R. and Dunlap, F. L.
1925. The production of experimental test biscuits and their volume measurement. Cereal Chem. Vol. II, pp. 87-94.

Jacobs, B. R.
1922. Self-rising flour—what is it? Am. Food J. Vol. 7, pp. 9-11.
Moher, L.
1923. Self-rising flour. J. Am. Assoc. Cereal Chem. Vol. 8, pp. 69-72.
Smith, F. C. and Bailey, C. H.
1923. The effect of chemical leavening agents on the properties of bread. J. Am. Assoc. Cereal Chem. Vol. 8, pp. 183-194.
Warning, W. G.
1923. Method of titrating calcium acid phosphate. J. Am. Assoc. Cereal Chem. Vol. 8, p. 74.

EFFECT OF TIME OF IRRIGATION ON PRODUCTION OF CRUDE PROTEIN IN WHEAT

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(Read at the Convention June 11, 1926)

In 1920 the Colorado Experiment Station undertook an investigation to discover if possible the most critical period in the demands for water in the development of the wheat crop. In the language of irrigation farmers, we were attempting to find out the best time to irrigate. While we realize that the best time to irrigate is when we have the water, we believe that if there were a choice in the use of water, a more economical use might be made if we knew the growth period at which water gave the largest results. After considerable preliminary work, the growth periods selected for application of water were germination, tillering, jointing, heading, blossoming, and filling. In addition to applications of water at these periods, one series of plots was irrigated at each of these periods. This we call "distributed irrigation." One series received no irrigation whatever. One received just sufficient irrigation to keep growing, and one was given two irrigations. Every treatment was replicated five times, so that results obtained are based upon averages of at least five different plots receiving the same treatment.

Our experiences in the first year showed that it was necessary to give a small irrigation at the time of planting in order to insure germination. Accordingly a 1-inch application of water was given to all plots at planting time. The total amount of water applied to each plot receiving treatment was the same, 7 inches—1 inch at planting time and 6 inches at one of the growth periods. Distributed irrigation received an application at each of the growth periods, giving a total of 7 inches applied.

The crop from each of these treatments has been analyzed. The analytical results, computed to a moisture-free basis, are shown in Table I.

TABLE I
EFFECT OF TIME OF IRRIGATION ON PRODUCTION OF CRUDE PROTEIN IN WHEAT
Computed to moisture-free basis.

Growth period at which irrigation was made	Crude Protein (N X 5.7)					
	1921	1922	1923	1924	1925	5-year average
	%	%	%	%	%	%
Germination	20.35	20.56	19.13	20.19	20.20	20.09
Tillering	21.89	21.09	19.48	20.00	20.90	20.67
Jointing	21.44	20.93	19.41	19.96	20.80	20.51
Heading	20.96	20.54	19.75	19.89	20.43	20.31
Blossom	20.75	20.82	20.06	20.19	20.55	20.47
Filling	21.30	20.90	19.90	19.92	19.37	20.28
Distributed	21.01	20.55	19.60	19.64	20.35	20.23
No irrigation	20.96	20.82	19.13	19.66	19.29	19.97
Keep growing	20.84	20.49	18.86	18.84	19.34	19.67
Two irrigations	.....	.....	19.28	20.46	20.25	19.996

A study of the crude protein (nitrogen × 5.7) indicates that irrigation at germination is slightly lower in protein production than irrigations at tillering and jointing. The irrigations at tillering and jointing produce the highest protein content in the crop. Irrigations at tillering are slightly higher than those at jointing. The lowest production of protein from one irrigation applied at any of these periods is from an irrigation at the filling period. Distributed irrigation, that is, a 1-inch application at each of the critical periods, gives a still lower average protein production. Where no irrigation was given, the protein content was lower than for any of the period applications. The only explanation we can offer is that there is not enough water to make development.

The keep-growing irrigation and two irrigations are essentially alike and all are lower than for the irrigation at a particular period. We do not offer any explanation for this behavior, simply record the tendency.

A study of Table I will indicate that the production of protein is not the same for all years, 1921 to 1925, but the tendency is the same and remains the same, and the average remains the same.

Table II shows the average production of protein for the 5-year period, 1921-25. While the production of protein is higher for irrigation at the earlier growth periods, the best quality of protein and the best quality of wheat are produced with irrigations at heading and blossoming periods. If not more than one irrigation is possible, an irrigation at the heading period is the

most important in the production of quality and yield. The total protein produced is slightly lower, but better grain and better quality of protein result.

TABLE II
EFFECT OF TIME OF IRRIGATION ON PERCENTAGE OF CRUDE PROTEIN IN WHEAT

	5-year average
Germination	20.09
Tillering	20.67
Jointing	20.51
Heading	20.31
Blossom	20.47
Filling	20.28
Distributed	20.23
No irrigation	19.97
Keep growing	19.67
Two irrigations	19.996

I have not analyzed the data to see whether the total production of protein per acre was greater or less for these periods. We have the data to make such determinations, but have not analyzed them. It is apparent from the study of the kernels that the earlier irrigations, which give a higher protein content, produce kernels which are not so well filled or developed as irrigations at heading and blossoming periods. Apparently the higher production of protein is largely a relative one.

INTERNATIONAL CONFERENCE POSTPONED

The International Conference of Research Workers regarding Flour and Bread Production, which was to be held in Prague from October 10 to 12, 1926, has been postponed to January or February, 1927, according to a cablegram received from Prague. Further information will be published when the details of the plans have been received from those who are arranging this conference.

A SUPERIOR NEW WHEAT FOR WESTERN AGRICULTURE

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The quest for superior wheat vareties has taken on somewhat more definite form since the beginning of the Twentieth Century. After the knowledge of Mendel's law became widespread, wheat breeders saw the possibility of bringing into combination certain qualities that exist in several wheats. They have, in a measure, come to see the importance of the milling and baking characteristics of wheats as well as the mere ability to produce good yields. Both the development of dependable methods for testing wheats for their milling and baking qualities, using only a few pounds, and the premiums or discounts paid by millers for superior or inferior varieties, have emphasized the necessity that is felt by millers for wheats of good bread-making quality. Altho in the details of both milling and baking tests there is much that is empirical, it can be confidently asserted that the methods possess a fairly satisfactory degree of accuracy, and that they differentiate well the inferior from the superior wheats.

As regards the purposes for which they are used, the wheats grown in the United States may be grouped into four groups, which intermerge to some extent—hard wheats used mainly for bread; soft wheats used for biscuit, cake, and pastry purposes and to some extent in mixture with hard wheat for bread; durum wheats used mainly for making semolinas to be manufactured into edible pastes, as macaroni, fettucelle, noodles, etc.; and feed wheats, principally emmer, used for animal feeding.

The most urgent demand is for more and better bread wheats. The word "hard" is applied in this country to certain spring and winter varieties of common wheat, *Triticum vulgare,* not, as in European countries, to *Triticum durum*. The qualities desired in a "hard" or bread wheat may be conveniently put into three classes: productivity, high flour yield, and power to produce a flour of high bread-making quality. As factors making up these characteristics and independent or more or less related to each other may be listed several qualities. High productivity is dependent upon a number of qualities, including disease resistance,

height and stiffness of straw, earliness, drouth resistance, non-tendency to shatter, winter hardiness, and many others which are confessedly somewhat vaguely known and which it is not within the province of this paper to discuss, tho one may merely refer to the difficulties of obtaining dependable figures with the variations occurring in yields produced by climatic influence, and also the effects of rotations, soils, and fertilizers.

High flour-yielding ability is an important varietal characteristic that is dependent upon the shape, density, and size of kernel; thickness of the bran coats and germ; and moisture content, these in turn being dependent upon several varietal and climatic factors. Some of the newer wheat varieties of widest popularity show consistently lower flour yields than older varieties which they have largely displaced. Marquis is one such, Bluestem and Fife having thinner skin than Marquis.

In the matter of bread-making quality, a fairly close correlation exists between high protein, high gluten content, and baking strength, the last being understood as ability to produce a loaf of large volume. Other qualities wanted in a bread-making flour are: a gluten of good, stable, elastic quality; high water-absorbing capacity; high bread-yielding ability; a dry, sound, slightly creamy white (not grayish or reddish) flour due to the vitreous character of the flour cells of the endosperm.

Many other related characteristics might be named, but in the writer's judgment the above comprise practically all important unit characters existent in wheat.

It is the purpose of this paper to set forth how remarkably high in most of these specifications the wheat to be described has been found to be.

The types and varieties of wheat grown in the western and intermountain states are for the most part of the softest character, high in flour yield but low in gluten, and having as a group the lowest baking strength of any wheats grown in the United States. They also possess low water absorbing capacity with consequent low bread yield. Large amounts of high-protein wheat from the northern and central Great Plains are shipped west to supply the needs for strong flour for bread making. The western wheats, however, have, as a group, a sufficient degree of baking strength for such goods as crackers and pies, but usually not enough for such yeast-raised or leavened goods as cakes or baking powder biscuits.

From time to time efforts have been made to find wheats of high baking quality, as evidenced by the large number of wheat varieties now grown in the intermountain and western states. Chul wheat, brought into prominence by Shaw, in California, is one instance. Hard Federation, Early Baart, and Burbank Quality are other varieties which possess merit in some degree over other western wheats, but relatively small in comparison with these wheats and in no case putting them into the class of hard bread wheats such as Turkey winter and Marquis spring. Early Baart, for which Clark, Martin, and Ball, in Bulletin No. 1074, United States Department of Agriculture, "Classification of American Wheat Varieties," gave as synonymous names Arizona Baart, Columbia, White Columbus, Diener No. 18, and Diener Hybrid, was first distributed for commercial growing by the Arizona Agricultural Experiment Station. It seems first to have been grown in the Orange River Colony or the Transvaal in South Africa; then to have been taken to Australia, where it was grown by a few farmers only in a small way. In 1900 the United States Department of Agriculture received some of it from Australia and from the office of Cereal Investigations it was taken to Arizona. By 1914 it was well established there and is now grown to a considerable extent in most of the western states, particularly the Pacific Coast states. Early Baart is a spring wheat, tho usually sown in the early winter.

In the Howard Laboratory analytical and physical testing methods have been developed, selected, and standardized for evaluating wheats strictly on their merits for bread making. The combination of practical and chemical tests used includes those which several decades of contact with and application to mill and bakery problems have yielded. A system of value scoring by assigning points of value to each item of the tests and analyses was carefully worked out and with minor modifications has been used in scoring for their milling and baking value wheats from the National Corn Exposition of 1908, and since then numerous wheats from state and Canadian provincial experiment farms, grain inspection departments, seedsmen, grain growers, and millers. It is believed that the relative values thus found express with reasonable accuracy the intrinsic milling and baking values of wheats.

Among the wheats which have been tested for the Arizona station, grown under the direction of Professor W. E. Bryan, a number of varieties have been found of superior milling quality, especially one designated as No. 24.

This wheat is a pure-line selection of a single head of wheat from selected hard strains of Early Baart. At the Arizona station several pedigreed strains of Early Baart have been grown and tested since before 1917. In 1920 a small amount of strain No. 34-14 was planted, and when the grains from these were examined it was noticed that a few of these were hard and vitreous in appearance while the rest had the usual soft texture of Early Baart. These hard kernels were planted separately and 19 plants were grown. Of these, 10 plants contained hard grains only, the rest being soft or mixed hard and soft. The heads all had the typical Baart appearance and came out at about the same time. These 10 hard strains were kept separate and multiplied. They have maintained their vitreous texture altho grown each year since then under irrigation, and have given as good wheat yields as the soft Baart. It is believed that they are part of the progeny of the original Baart carried along as an unnoticed admixture with the typical soft, commercial type, assuming that the variety came from a cross between a hard and a soft variety and that it was distributed while it consisted of mixed, unfixed grains, both hard and soft. In milling and baking tests, 10 of these hard strains, while not more notable for higher baking strength than the soft Baart, showed decidedly higher water absorption (6%) and gluten (2%).[1]

There are now about 80 strains of hard Baart which have been grown for 5 years, some of them both in small plots and in field plots and always with as good yields as the softer varieties. No. 24 has been grown for three years and stands out in the milling and baking tests ahead of all the others.

Altho the new variety was first thoroly tested in 1925, there is every reason to believe that the outstanding combination of high qualities which the tests reveal will be maintained, since all the wheats tested were grown and milled and the flours were stored and baked under approximately identical conditions. It cannot be expected that in all years exactly the same degrees of quality will be exhibited, but there can be no doubt that this wheat, under the same general soil and climatic conditions in which it has shown its superiority, will continue to show its outstanding merit. A variety possessing so many superior traits in combination is potentially of enormous value to the farmers of the Southwest and hence to the millers and to the whole nation.

[1] Bryan and Pressley "Hard grain texture as a basis of selection for improving the quality of early Baart wheat," Jour. Am. Soc. Agron., Vol. 17, No. 7, 440ff.

The kernels of this wheat have the typical hard and vitreous appearance of an amber durum, not the duller pale yellow of common white wheats. In this respect it is deceptive. At the International Hay and Grain Show held at Chicago, November 28 to December 5, 1925, it was taken by the judges to be "purely a durum wheat." The plant characters are those of Early Baart wheat, not those of the durums. It is bearded, with rather short awns, smooth chaff, white, long, and narrow. The grain has a narrow, shallow crease with rounded cheeks. The grains are large and nearly uniform in size and color, elliptical, symmetrical, with a medium-sized to small germ.

The sample of the grain on which the tests referred to below were made weighed 58 pounds per bushel. The grain is held fairly closely in the head, but not so tightly as to make threshing difficult. It has shown very little smut damage.

This wheat in 1925 produced 55 bushels per acre on 200-foot rows replicated five times. On a 40-acre field, the average yield of the original soft Baart was 42 bushels. Discounting the 200-foot row yields by 20 per cent, the results are fairly near each other. Forty-two to 44 bushels is a very excellent showing. The average yield of irrigated wheat in Arizona is 30 bushels per acre, or about double the yield in the United States as a whole. The flour produced is in none of its characteristics like flour from durum wheats. It has not the yellowish white color characteristic of flours from amber durums and, to a lesser degree, of club wheats, nor has it the chalk-white color of many western white wheats. The flour has the moderate tinge of creaminess usually found in soft eastern winter wheat flours. In baking strength, it is well above the best of the durum flours which show relatively high baking strength; its baking strength, in fact, is greater than the average hard red winter wheat from Kansas.

The results of the milling and baking tests, the analyses and the scoring values assigned to this wheat and a few others are shown in the table. No. 1 is a soft strain of Early Baart from which No. 24 was selected. It is fairly representative of the better common wheats of the west; No. 38 is a hybrid between Turkey and Sonora; No. 41 is Hard Federation, a selection from Federation, a variety produced by Farrer, the Australian wheat breeder. The "standard spring" is an average of northwestern grown spring wheats grading "No. 2 dark northern" and is used as the standard of comparison and assumed to be worth $1, or 100 per cent.

Results of Tests

	No. 24	No. 1	No. 38	No. 41	Standard spring
	Tests on the Wheat				
Moisture, %	8.1	7.2	8.5	6.8	12.2
Ash, %	1.57	1.69	1.89	1.74	1.82
Protein, %	15.83	12.65	10.62	11.24	12.60
Cleaning loss, %	0	0	0	0	2.0
Milling yield, %	79.5	73.5	72.0	76.8	72.0
	Tests on the Straight Flour				
Acidity, %	0.086	0.072	0.079	0.111	0.100
Soluble carbohydrates, %	3.1	1.5	1.5	2.7	1.3
Dry crude gluten, %	13.7	10.1	9.1	9.6	11.9
Quality of gluten	Firm elastic	Stiff elastic	Firm elastic	Firm elastic	Elastic
Color	1.5	1.5 Good	2 Good	2 Good	2 Good
Color quality	White creamy dull	White little dull	Creamy white dull	White creamy grayish	Creamy white dull
Color of crust	Light brown	Light brown	Light brown	Light brown	Light brown
Volume of loaf, cu. in.	192	160	134	135	198
Shape	Normal	Normal	Normal	Slight crack on top	Normal
Texture	Normal	Normal	Slightly coarse	Coarse	Normal
Odor	Normal	Normal	Normal	Normal	Normal
Weight of loaf, oz.	18.38	17.25	18.13	17.88	17.44
Water used, oz.	7.94	6.69	7.50	7.31	6.88
True absorption, %	66.2	55.7	62.5	60.9	57.3
Bread per barrel, lb.	300.2	281.8	296.1	292.0	284.9
Relative value	$1.214	$1.046	$0.944	$0.945	$1.00

In the table, protein represents nitrogen times 5.70; cleaning loss is the percentage of screenings and dust removed by the milling separator and scourer. After scouring, the wheats were tempered with water so as to bring the water content nearly to 15 per cent, and allowed to stand over night in tin cans. They were then milled by our milling system, which follows closely the milling system of a four-break mill. The yields are the percentages of straight flour as calculated from the cleaned wheats used and are fairly accurately comparable to yields obtained in merchant mills. The flours were stored in loosely covered tin pans and allowed to age for 8 weeks before the baking and other tests were made. Straight flours thus made and stored show practically the same results as straight flours made in merchant mills.

The acidity test (calculated as lactic acid) showed low figures. The acidity is a fair measure of the degree of soundness, any increase over the normal limit, which for straight flours is about 0.125 per cent, showing the degree of unsoundness, if any. The soluble carbohydrates of these flours are normal, soluble car-

bohydrates or sugars and dextrins being the fermentable materials and in flours from sound wheats seldom exceeding 6 per cent in amount.

The gluten tests were made using all precautions necessary for uniformity and to prevent losses during washing.

The baking tests were made by the Howard Laboratory standard baking method with controlled conditions including temperatures, yeast, amount of mixing action, etc. Color grades were marked by the Howard system. Standard patents grade 1.5 (good, medium, and minimum) and straights 2 (good, medium, and minimum). The average volume of northwestern spring wheat patents and straights of the 1925 crop was 205 cubic inches, and the weight of loaf and water used 17.75 and 7.25, respectively, for fresh-milled flours, older flours increasing, of course, above these figures in absorptions and bread yields. It should be noted that these figures for loaf volume, weight, and water used are higher in the flours of the 1925 crop of springs than in the standard spring average shown in the table. This standard is an average of 10 years' inclusions of spring wheats and the straight flour milled therefrom. The volume of the test loaves is a measure of the true baking strength, indicating the ability of the flour to expand, hold up well, and give a light, well-piled loaf. If the test loaf has large volume, the flour possesses a good degree of reserve strength. Such flours, when used in bread making in the household or bakery, will in the dough and loaf, if necessary, stand a much greater degree of unfavorable treatment without spoiling the lightness of the bread than will flours showing lower loaf volumes in the laboratory test. It is in baking strength that No. 24 excels.

The weight of loaf is recorded as it comes from the oven. It shows the ability of the flour to hold the absorbed water and make a good bread yield to the barrel of flour. The water used shows the actual absorbing and retaining capacity of the flour in baking. Here, especially, the flour from this wheat shows its preeminence among a group of flours of extraordinary absorptions.

The results of the tests of No. 38 and also of No. 41 may be taken as fairly representative of Pacific Coast common wheats, while No. 1 is typical of the better western wheats with a moderate degree of baking strength.

It can confidently be asserted that this wheat, if grown under the same conditions as prevailed, will be superior in baking strength to all other types except spring, and will equal many

springs in this respect. Because of its extraordinary flour yield and its very high water absorption and gluten content as well as its good color, in which it is only excelled by No. 1, and because of other good qualities, this wheat is one which millers will gladly pay high premiums for.

How it will behave under varying conditions of soil and climatic differences and without irrigation can be shown only by further tests, but it is reasonable to believe that its outstanding merits will continue to assert themselves in comparison with other wheats which have been tested under the same conditions.

In growing these wheats at Mesa, the land is irrigated by giving from 8 to 10 acre-inches just previous to seeding, which is done about December 1. It is again irrigated when the grain is in the boot, giving about 6 inches of water, and, finally another 8 inches when the wheat is in the soft dough stage. In addition, about 3 inches of rain falls, on the average, before the grain has reached the hard dough stage.

The tests made on this wheat demonstrate conclusively its outstanding merits as a superior bread-making wheat for western arid and semi-arid conditions.

The factors which contribute to the high quality of this wheat are, first, and by far the most important, its inherent heritable high quality; second, the fertile soil upon which it was grown, especially high in nitrogen; third, the climatic conditions both natural and artificial, of which the control of water applied during the ripening stages of the wheat is important. All these conditions are important, and without each, the best results could not be obtained. Nitrogen in the soil is considered by Professor Bryan to be the first limiting factor, old alfalfa sod being probably the best ground for quality wheat. In this he agrees with Jones, Colver, and Fishburn, of the Idaho Experiment Station,[2] who found that with a proper balance of soil fertility, wheat of very high quality could be grown there, but when sagebrush land was brought into cultivation, it could not produce wheat of good quality for bread making regardless of the variety grown because of its deficiency in nitrogen, never having been improved by alfalfa or other nitrogen-gathering legumes.

The widespread culture of this wheat throughout the west will solve the problem of a high-gluten bread wheat for the west and add value to all the agricultural land of the west, both irrigable and dry.

2 Jones, Colver and Fishburn. J. Agr. Sci. Vol. 10, pp. 290 ff.

It is planned to continue the tests and then multiply the seed until 50 bushels are obtained, then to turn this seed over to the Farm Bureau Pure Seed Association for distribution among its members. The wheat obtained from these planters will thus be under the control of the association, so that adequate distribution can be had for it without any commercial concern obtaining a monopoly on it and selling it at exorbitant prices.

The writer wishes to acknowledge gratefully the co-operation and assistance of Professor Bryan in the preparation of this paper.

PLASTICITY—ITS POSSIBILITIES IN CEREAL RESEARCH

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(Read at the Convention, June 11, 1926)

If a perfectly elastic solid be subjected to a shearing stress, a certain strain is developed which entirely disappears when the stress is removed. The total work done is zero, the process is reversible, and viscosity can play no part in the movement. This is not a case of *flow,* but of elastic deformation. If a body which is imperfectly elastic as regards its deformation be subjected to shearing stress, it will be found that a part, at least, of the deformation will remain long after the stress is removed. In such a case, work has been done in overcoming some kind of internal friction. We may distinguish the different kinds of flow under three regimes.

It is characteristic of *viscous* or *linear* flow that the amount of deformation is directly proportional to the deforming force; and the ratio $\frac{\text{deforming force}}{\text{deformation}}$ gives a measure of viscosity. The question has been raised as to whether or not this ratio is truly constant, but it appears that only one qualification is necessary. In very viscous substances, time may be necessary for the flow to reach a steady state (aside from any period of acceleration) because with substances like pitch the viscous resistance develops slowly. Therefore, the ratio of force/deformation gradually increases when the load is first applied. Even in this case, the ratio gradually reaches a value which is independent of the amount of the load. However, as the deforming force is gradually increased, a point may be reached where the ratio suddenly decreases. At this point, the regime of *turbulent* or *hydraulic* flow begins.

There are substances, on the other hand, for which the value of the ratio increases indefinitely as soon as the deforming force falls below a certain minimum; since the deformation approaches zero quite rapidly as the deforming force is further decreased. These substances are said to be *plastic*. In the third regime—that of *plastic flow*—it is generally understood that a definite shearing force is required before any deformation takes place. Whether or not this is *strictly* true has not yet been definitely established.

The viscosity of a substance is measured by the tangential force on a unit area of either of two horizontal planes at unit distance apart, required to move one plane with unit velocity in reference to the other plane, the space between being filled with the viscous substance. The ratio of force/rate of shear ($\frac{F}{V}$) is called the coefficient of viscosity—usually denoted by the symbol η. The coefficient of fluidity is equal to the reciprocal of this value. That is: $\Phi = \frac{1}{\eta}$. The coefficient of fluidity may be defined as the velocity of the two unit planes with respect to the force applied.

Many types of instruments have been designed to measure viscosity, but most of them merely give relative values. These relative values, however, may be expressed in absolute units (Poise) by calibrating the instruments by means of substances of known viscosity. There are three classes of viscometers: (a) those measuring the resistance offered to a moving body by a viscous fluid; (b) those measuring the rate of flow; (c) those making use of decay of waves, oscillations, or vibrations of surface or upon rate of crystallization.

The law of Poiseuille is as follows: The amount of fluid which transpires in a given time and at a definite temperature is directly proportional to the pressure and inversely proportional to the length of the capillary. That is: $\eta = K \frac{P}{L}$. Viscosity (at least for liquids) is evidently a definite physical quantity, since results obtained in different ways seem to lead to the same value. The absolute unit (Poise) was named in honor of Poiseuille, who did so much work in the development of viscometric studies. The ratio of $\frac{\text{volume of liquid}}{\text{time}}$ to the shearing stress, in the case of viscous liquids, is a straight line function, passing through the origin. This is not the case with plastic solids. Therefore, the shape of the flow-shear curves serves to distinguish between

liquids and solids, just as sharply as liquids and gases are differentiated at the critical temperature and pressure.

Plasticity of Solids

Only by the behavior of materials under shearing stresses are we enabled to distinguish between a fluid and a solid. If a body is continuously deformed by a very small shearing stress, it is a fluid; whereas, if the deformation stops increasing after a time, the substance is a solid. This distinction is *theoretically* sharp like the distinction between a liquid and a gas at the critical temperature, but just as a liquid may be made to pass into a gas insensibly, so a solid may grade insensibly into a liquid. Glass and pitch are familiar examples of very viscous liquids. Paint, clay-slip, and thin mud, in a similar manner, must be classed as soft solids. According to the experiments of Bingham and Durham (1911) the concentration in which the fluidity becomes zero under a very small shearing force serves to demarcate the two states of matter.

This simple distinction is not always sharply drawn nor is its significance thoroly appreciated; and for this reason much labor has been ill-spent in the attempt to measure the viscosity of solids, on the assumption that solids are only very viscous liquids and therefore that plasticity and the fluidity of solids are synonymous terms. The results are unintelligible because the determination of viscosity by various methods seldom leads to the same value.

The views of Maxwell (1916) expressed in his "Theory of Heat" are especially noteworthy and are quoted at length:

"If the form of the body is found to be permanently altered when the stress exceeds a *certain* value, the body is said to be soft or plastic and the state of the body when the alteration is just going to take place is called the limit of perfect elasticity. If the stress, when it is maintained constant, causes a strain or displacement in the body which increases continually with the time, the substance is said to be viscous.

"When this continuous alteration of form is only produced by stresses exceeding a certain value, the substance is called a solid, however soft it may be. When the very smallest stress, if continued long enough, will cause a constantly increasing change of form, the body must be regarded as a viscous fluid, however hard it may be.

"Thus a tallow candle is much softer than a stick of sealing wax; but if the candle and the stick of sealing wax are laid horizontally between two supports, the sealing wax will, in a few weeks in summer, bend under its own weight, while the candle remains straight. The candle is, therefore, a soft (or plastic) solid, and the sealing wax is a very viscous liquid.

"What is required to alter the form of a soft solid is sufficient force, and this, when applied, produces its effect at once. This is, of course, only relatively true, because plastic deformation is a function of the time. In the case of a viscous fluid, it is time which is required, and if enough time is given the very smallest force will produce a sensible effect, such as would be produced by a very large force if suddenly applied.

"Thus a block of pitch may be so hard that you cannot make a dent in it with your knuckles; and yet it will, in the course of time, flatten itself out by its own weight and glide down hill like a stream of water."

We may now define plasticity as a property of solids by virtue of which they hold their shape permanently under the action of small shearing stresses; but they are readily deformed, worked, or molded, under somewhat larger stresses. Plasticity is thus a complex property, made up of two independent factors which we must evaluate separately.

In a plastic solid, a certain portion of the shearing force is used up in overcoming the *internal friction* of the material. If the stress is just equal to the friction or yield value, the material may be said to be at its elastic limit. If the stress is greater than the friction f, the excess, F-f, will be used up in producing plastic flow according to the formula: $v=\mu\ (F-f)r$ where v=velocity of flow, r is the distance between planes, and μ is a constant which we will call the *coefficient of mobility* in analogy to the fluidity of liquids and gases. If we were to plot the volume of flow per unit of time against the shearing stress, we would again obtain a straight line for a given material but it would not pass through the origin.

It is easy now to see why the "viscosity" of plastic substances, as measured in the usual way for liquids, is not a constant.

When the stress applied is not equal to the yield value, the material undergoes elastic deformation and an opposing force arises which would restore the body to its original shape if it were perfectly elastic, as soon as the stress is removed. On the

application of the stress, the restoring force is first zero, then gradually increases to a maximum, when at last the flow causes the strain to disappear as fast as it is produced.

To determine the two quantities, friction and mobility, which go to make up the plasticity of a material, it is necessary to make at least two measurements of the flow, using different stresses. We may use the tube method of Bingham (1922), the torsion method of Perrott and Thiessen (1920), or we may observe the flow in a rod under traction or torsion, the flow of a cylinder under axial compression, the rate of bending of a horizontal beam of the material under its own weight, or the flow of a freely descending stream of the material. Lord Kelvin (1865) has suggested another method based on the decay of vibrations in solid bodies.

The friction is most easily obtained by extrapolating the curves of flow-rate/stress until they intersect the stress axis. All curves for a material should intersect just as they cross this axis, giving the same value for the friction of the material in question.

While the flow-rate/stress curves should be straight lines, we seldom obtain them as such in practice. At the present time, there is no good explanation for the discrepancies. Therefore it is common practice to choose our experimental conditions so that the deviations are reduced to a minimum. Buckingham (1921) has offered two correcting factors to take care of slippage and seepage. If the plastic material has a lower concentration of solid at the section near the wall, the friction would be reduced, and the flow observed would be higher than it ought to be. This error is called slippage. If the shearing stress causes the liquid to flow through the network of solid particles, the flow observed would also be higher than it should. This error is called seepage. Buckingham also suggests that the friction between molecules, when the material is flowing, may differ from the friction when in the static state. Bingham (1922) has pointed out that the friction will probably depend also on the size of particle.

The Bingham and Murray (1923) combined viscometer and plastometer is simple in construction and seems well adapted for use with flour doughs. The theory of its operation is as follows: When a substance enters a capillary (empty) under a given head, the shearing stress continually changes, the flow becoming slower as the shearing stress decreases. Since this shearing stress may be calculated mathematically, it is only necessary to measure

the lengths of time required for the material to reach successive points in the capillary, in order to obtain sufficient data for viscosity and plasticity calculations.

In very accurate work, a chronograph is used by means of which the time can be read accurately to one hundredth of a second. Ordinarily, work may be carried on by means of a good split-second stop watch. The temperature is kept constant with only a deviation of a small fraction of a degree by means of a water thermostat. The volume of flow at each of the marks on the capillary is accurately determined by calibrating with mercury. The radius of the tube is also estimated. The time is read experimentally. The stress at each point is obtained by integrating the difference of stresses at both extremities of the tube.

$$F = \frac{1}{L_2 - L_1} \int_{L_1}^{L_2} \frac{PR}{2L}\, dL = \frac{PR}{2(L_2 - L_1)} \log_e \frac{L_2}{L_1} = \frac{PR}{20} \log_e \frac{L_2}{L_1}$$

[when $L_2 - L_1 = 10$ cm.]

$$F = 0.1151\ PR \log_{10} \frac{L_2}{L_1}$$

With a material as plastic as flour dough, the error due to seepage should be negligible. The slippage error will depend on the material used in the capillary construction. The slippage value will depend on whether or not the medium (water) wets the walls of the capillary.

Theory of Plastic Flow

A plastic solid is made up of particles which touch each other at certain points. The spaces between the particles may be empty or filled with a gas, liquid, or amorphous solid. Flow necessitates the sliding of the particles over one another according to the laws of friction, so long as the particles are large enough to neglect Brownian movement. It is not necessary that the particles be close packed. As a matter of fact, this would prevent flow from occurring. It is merely necessary that the particles form arches capable of carrying a load. If the concentration is increased to the point that this arching is continuous throughout the mass, the system becomes a solid and the friction will have a finite value. Only when the pore space exceeds 50%, do we have a value of flow which is finite. There is also a maximum value for the pore space. When this value is exceeded, the system ceases to be a solid. As the diameter of the particles is *decreased,* the opportunity for the particles touching is increased, which increases the friction. This

also has a limiting value where the correction for Brownian movement becomes appreciable. Adhesion plays a rôle. Therefore, the concentration of hydrogen-ions is important. Colloids also have a noteworthy effect, therefore the presence of electrolytes is important. The mobility depends on the fluidity of the medium. This is, in turn, affected by the temperature. Flocculation and deflocculation also have an effect. This also calls for control of hydrogen-ion concentration.

It is quite evident, therefore, that while we may make use of plasticity values, it may be a long time before we can derive a theoretical equation for calculating such values.

The statement has already been made that when viscosities are run on materials that are plastic, we seldom get values that are constant. Sharp (1926) has already shown that flour-in-water suspensions cease to be viscous liquids and become plastic when the concentration exceeds a certain value. For average flours, this value is approximately 9-10 per cent by weight, figured on the dry basis. Sharp's conclusions have been corroborated in the Pillsbury laboratories at Minneapolis, where a Bingham and Murray type plastometer is used. Consequently, since it is apparent that flour-water doughs are plastic systems, investigation along the line of plasticity determinations seem logical and amply justified.

Before setting out on such investigations, it is necessary to investigate the effect of each of the variables on the final result. These variables include concentration; particle size; temperature; radius of capillary; hydrogen-ion concentration; method of mixing; previous treatment, such as time of standing; and anything else which will affect the colloidal state of the dough. When the effect of each of these variables has been ascertained, a procedure can be outlined which will tend to reduce the experimental error to a minimum.

It is a comparatively easy task to keep the temperature, pressure, hydrogen-ion concentration, time of standing, and size of capillary constant. But it is not so simple a task to keep the method of mixing, previous treatment, particle size, and such variables as influence the colloidal nature of the system at a constant value. For example, if we try to standardize the particle size by bolting through various sizes of cloth, we find that the degree of refinement as measured by ash is different in each fraction so obtained. Moreover, in most cases the analysis of the sample is quite different. The amount of ruptured particles, both starch and protein, will be

larger in the finer samples than in the coarser fractions. There is evidence that a homogeneous sample of wheat can be milled in several separate runs and milled to the same ash, and yet there will be different characteristics to each run, owing to variation of milling conditions, etc.

If the electrolytes are extracted by a method similar to that of Sharp and Gortner, every effort must be made to keep the conditions of extraction uniform. The time, amount of water used, and particularly the temperature of extraction must be maintained constant. However, these precautions are in nowise more necessary in plasticity determinations than they are in any other type of physico-chemical investigation.

As regards the practical application of plasticity values to flour manufacture, too little data are available at the present time to enable us to say whether or not there will be correlation between the baking value of a flour and its plastic values. This much is true—a dough made from wheat flour is a plastic system, and any investigation of its physico-chemical properties should take this fact into account. Plasticity determinations give definite results for each flour if conditions are held strictly constant. Whether or not these physical constants can be correlated with practical tests which give a measure of flour quality, such as the baking test or the expansion test, remains to be seen. Investigations of such a nature are being carried on in the Pillsbury laboratories, and it is probably only a question of time before information will be available which will enable other cereal laboratories to investigate the plastic characteristics of flour doughs. Plasticity determination is proving of value in other industries which have plastic material to deal with, such as the rubber industry and the paint industry. Let us hope that it will also prove its worth in our own industry, which has long felt the need of a physico-chemical measure of that elusive will-o'the-wisp—"Gluten Quality."

Literature Cited

Bingham, E. C.
1922. Fluidity and plasticity. McGraw-Hill Co., N. Y.

Bingham, E. C. and Durham, T. C.
1911. The viscosity and fluidity of suspensions of finely divided solids in liquids. Amer. Chem. J., Vol. 46, p. 278.

Bingham, E. C. and Murray, H. A. Jr.
1923. A new combined viscometer and plastometer. Research Pub. of Lafayette College, Serial No. 8.

Buckingham, E.
1921. On plastic flow through capillary tubes. Proc. Am. Soc. Test. Materials, Vol. 21, p. 1154.

Maxwell, J. Clerk.
1916. Theory of heat. Longmans Green & Co., N. Y.

Perrott, G. St. J. and Thiessen, R.
1920. Carbon black—its properties and uses. J. Ind. Eng. Chem. Vol. 12, p. 324.

Sharp, P F.
1926. Wheat and flour studies V. Plasticity of simple flour-in-water suspensions. Cereal Chem. Vol. III, p. 40.

Thomson, Sir Wm. (Lord Kelvin)
1865. On the elasticity and viscosity of metals. Phil. Mag., Vol. 30, p. 63.

SHOULD FLOUR BE ARTIFICIALLY MATURED AND DECOLORIZED?

By M. Javillier

(Summary of a translation from the French by Alma E. Warthen, Bureau of Chemistry, U. S. Department of Agriculture.)

Millers of France are now limited to a single type of flour, the straight grade. This was not the case before the war, when variable rates of extraction were produced. At that time the color of the flour was of more importance than at present.

Agents used for decolorizing flours have been found to induce the maturing of the flour which is an advantage, a convenience, and a material gain. An antiseptic effect is also exerted by the bleaching agents.

Chlorine mixed with air is used at the rate of 14-18 grams of chlorine per 100 kilograms of flour. Nitrogen chloride, NCl_3, is used in proportions varying from 3 to 25 grams per 100 kilograms. In the Andrews process, 100 to 200 cc. of nitrogen peroxide is used per 100 kilograms of flour. With the Stacey apparatus the nitrogen peroxide constitutes from 0.12 to 0.14 per cent of the oxidizing atmosphere, and 0.7 to 4.0 grams of nitrogen peroxide are used per 100 kilograms of flour. In the Novadelox method, 3.5 to 4.5 grams of benzoyl peroxide is used per 100 kilograms of flour. Nitrosyl chloride, NOCl, is also used in flour bleaching. Water probably plays a direct rôle in the reactions involved in the use of all these reagents. Thus

$$(1)\ Cl_2 + H_2O \rightarrow HCl + HOCl$$
$$(2)\ NCl_3 + H_2O \rightarrow NH_3 + HOCl$$
$$(3)\ 2NO_2 + H_2O \rightarrow HNO_3 + HNO_2$$
$$(4)\ NOCl + H_2O \rightarrow HNO_2 + HCl$$

Benzoyl peroxide is slowly catalyzed with the formation of atomic oxygen possessing great oxidizing activity.

Carbohydrates, including starch, sugars, and cellulose, are probably not affected by these bleaching reagents.